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MECHANICAL BEHAVIOR OF HIGH TEMPERATURE COMPOSITES

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MATERIALS DIRECTORATE
WRIGHT LABORATORY
AIR FORCE MATERIEL COMMAND
WRIGHT PATTERSON AFB OH 45433-7734

NOTICE

THIS TECHNICAL REPORT HAS BEEN REVIEWED AND IS APPROVED FOR PUBLICATION.

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THE OBJECTIVE OF THIS PROGRAM IS TO DEVELOP AN UNDERSTANDING OF CERAMIC COMPOSITES AND OXIDATION-PROTECTED CARBON-CARBON COMPOSITES, AND TO PROVIDE GUIDANCE FOR FUTURE MATERIAL DEVELOPMENT OF COMPOSITES BY THE EVALUATION AND CHARACTERIZATION OF THESE MATERIALS FROM 25 DEGREES C TO 2200 DEGREES C IN INERT AND OXIDIZING ENVIRONMENTS.

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PREFACE

This final report was prepared by Southern Research Institute for the Air Force under contract F33615-86-C-5005. Southern Research Institute was the prime contractor; General Electric Aircraft Engine - Evandale, Materials Sciences Corporation, United Technologies Research Center, and Virginia Polytechnic Institute were subcontractors. The tasks at Southern Research were performed in the Mechanics Research Department under the supervision of H. Stuart Starrett, Director. Terry R. Barnett was the project monitor and prepared the final report. The report was prepared for reproduction by Ms. M. M. Oliver. The contract was monitored by David Jones and Ted Nicholas at Wright Laboratory.

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1.0 INTRODUCTION

The objective of this contract was to develop an understanding of ceramic composites and oxidation-protected carbon-carbon composites, and to provide guidance for future material development of composites by the evaluation and characterization of these materials from 77°F to 4000°F (25°C to 2200°C) in inert and oxidizing environments.

The scope of the program included:

1. Initially evaluate state-of-the-art ceramic composites and oxidation protected carbon-carbon composites up to the maximum potential use temperature, or 4000°F, using available test and evaluation techniques.

2. Utilize mechanics models and theories to assess candidate materials, to analyze test and evaluation techniques, and to interpret and analyze material responses.

3. Improve and develop test methodologies for ceramic and oxidation protected carbon-carbon composites from 77°F to 4000°F in inert and oxidizing environments.

4. Investigate and develop air furnace and high temperature heating techniques to be used for oxidation testing and for mechanical and thermal property evaluation up to 4000°F.

Southern Research was the prime contractor and subcontracted with Materials Sciences Corporation, General Electric Aircraft Engines, United Technologies Research Center, and Virginia Polytechnic Institute and State University. The role of each contractor was as follows:

1. Southern Research Institute: Overall contract management. Development of test techniques for mechanical property measurements in air from 70°F to 4000°F. Develop techniques to evaluate a) tensile stress-strain response to 3200°F, without strain to 3500°F, b) compressive stress-strain response to 2800°F, c) interlaminar shear strength to 2800°F, d) Iosipescu shoar to 2800°F, e) flexural load-deflection to 2800°F. Generate mechanical and thermal property data packages on ceramic matrix and carbon-carbon composites. Development of 4000°F air furnaces with relatively short up and down cycle times for exposing small samples of materials from AF material development efforts to high temperature oxidizing environments.

2. Materials Sciences Corporation: Develop and understanding of stress - degradation mechanisms in ceramic matrix and carbon-carbon composites. This involves the demonstration of analytical models to correlate with generated experimental data.

3. General Electric: Configuration Assessment for critical properties in turbine engine components. Long-term mechanical and thermal property data on ceramic matrix and carbon-carbon composites.

4. United Technologies Research Center: Materials screening and physical property measurements. Develop an understanding of thermochemical degradation mechanisms in ceramic matrix and carbon-carbon composites which involves the development of facilities and test methodology for oxidation testing from 2800°F-3200°F.

5. **VPI & SU: Phenomenological models for prediction of long-term behavior of ceramic matrix composites.**

In the following sections, a summary of the results of the program will be described. Details of the work have been previously published and released in the form of subtask, interim, and contractor reports, and presentations made to the Air Force. A complete list of reports and papers is given in the closing summary section.

2.0 GE CONFIGURATION ASSESSMENT

GE was subcontracted to qualitatively define the critical, component-dependent material requirements that need to be addressed in order to integrate refractory composites (specifically carbon-carbon and ceramic matrix composites) into advanced man-rated turbine engine designs. The integrated candidate engine components included:

1) Combustor Liner
2) High Pressure Turbine (HPT) Vane
3) HPT blade
4) Low Pressure Turbine (LPT) Vane
5) Turbine Shroud
6) Turbine Frame Liner
7) Turbine Outlet Guide Vane
8) Centerbody
9) Flameholder
10) Augmentor Liner
11) Exhauser Liner

In a qualitative sense, it was found that these components share many of the same material requirements. These critical requirements were as follows:

1) Operating Temperature
2) Durability
3) Producibility
4) Thermal Conductivity
5) Thermal Shock Resistance
6) Thermal Fatigue Endurance
7) Specific Strength
8) Specific Stiffness
9) Erosion Resistance
10) Abradability

The assessment concluded that refractory composites do posses potential for gas turbine applications and virtually all hot section

components require materials with high operating temperatures, time at temperature, thermal shock, and thermal fatigue capabilities.

The details of this evaluation can be found in SRI interim report SRI-MME-90-393-6145 entitled "Mechanical Behavior of Composites at High Temperature," submitted to the Air Force in October 1990.

3.0 TEST METHOD DEVELOPMENT

3.1 Mechanical Tests Developed at Southern Research

Southern Research developed mechanical test techniques to evaluate candidate materials in oxidizing environments. The two basic test methods were tension and compression. From these two basic tests, the following tests have evolved:

1) Tensile Response to 3200°F in air
   a) Stress-Strain
   b) Fatigue
   c) Creep/Stress Rupture

2) Compressive Response to 2800°F in air
   a) Stress-Strain
   b) Fatigue
   c) Creep/Stress Rupture

3) Interlaminar Shear to 2800°F in air

3.1.1 Tensile Response to 3200°F in Air

The specimen configuration designed by Southern for thin laminates is given in Figure 3.1-1. The specimen has an overall length of 6.00 inches with a gage section width of 0.300 inches. (The gage width can vary based on the material orientation.) The three inch radius allows minimal stress concentration at the tangent; the transition region allows for smooth thermal gradients which is important in elevated temperature testing. Strain measurements are made over a distance of 1.0 inch in the test section. The configuration can be altered as material orientation and needs dictate. Further details of the specimen configuration are given in the figure.

Air tensile evaluations are performed in the 3200°F Air Tensile Set-up. Figure 3.1-2 shows the specimen heating technique. Specimens are radiantly heated by "U"-shaped silicon carbide elements. Fibrous zirconia

board provides thermal insulation. Power is supplied and controlled by a 5KVA variable transformer. The furnace and specimen are exposed to the laboratory atmosphere to provide an air test environment. Temperature is monitored by an optical pyrometer. Readings are taken through an open sight port in the zirconia board.

Strain measurements are made with clip-on extensometers which measure the elongation between two flags, shown in Figure 3.1-3. Ports with visual openings on opposite sides of the furnace provide a means of attaching two sets of clip-on arms to the flags. The flags are attached to the specimen by means of a 0.003 inch grove machined in the specimen or in ceramic paint coated on the specimen, Figure 3.1-4. The relative travel of the two flags provide a deformation signal and readout is continuous on a millivolt recorder.

Figure 3.1-2 also gives the specimen gripping technique. The action of the support wedges maintains alignment of the specimen in the load train. The ends of the specimen are machined flat and parallel; each end fits against a heat dam to control thermal gradients in the specimen. Specimens of variable thickness can be accommodated with this wedge system. The specimen and all components of the pullrod assembly system are machined to close tolerances to maintain alignment.

Tests are made using precision chain grip pullrods. The original grips were designed and developed at Southern Research as part of an in- house effort to aid ASTM Committee C-5 in the development of a tensile test for polygraphites as specified in ASTM C-749. For this contract, the design was further refined and a new set of grips is currently being used with the air tensile setup. The modified grips and pullrods minimize non-axial loading of the specimen through precision adjustors. The measured parasitic (bending) strain is on the order of ±10 μ". A routine alignment check is made to insure the parasitics are small. A schematic of the setup is shown in Figure 3.1-5.

Instrumentation for a typical test includes a 10,000 pound Strainsert load cell, constant d.c. voltage power supply, an X-Y recorder, and means to measure specimen temperature. The load cell receives a constant d.c.

voltage input from the power supply and transmits a millivolt signal (proportional to load) to the X-Y recorder. Simultaneously, the clip-ons transmit a signal (proportional to strain) to the recorder. Thus, a continuous plot of stress-strain is recorded.

Please note that the above discussion was limited to thin flat coupons for illustrative purposes only. The same techniques have been applied to testing of monolithic ceramics where specimens are generally cylindrical. These methods have been implemented by Southern Research for Allison Gas Turbine on DOE's "Life Prediction" of monolithic ceramics program. In the referenced program, high temperature tensile stress-strain and tensile creep response were the main interests.

The same methodology used for static tensile stress-strain has been extended to tensile fatigue and creep. Fatigue tests are performed with the air set-up in a servo-hydraulic machine and creep tests are performed in creep frames.

3.1.2 Compressive Response to 2800°F in Air

The preferred compressive configuration is given in Figure 3.1-6. This is commonly called the "bow-tie" design. The specimen is generally 3.00 or 4.00 inches long. Strain measurements are made over a distance of 0.900 inches in the test section. Further details are given in the figure.

In the mid 1980's, Southern designed a compressive test set-up to evaluate thin composites. The fixture was called the Controlled-Face-Supported (CFS) apparatus, Figure 3.1-7. The facility provides flat, lateral support for both faces along the entire length except for an unsupported region at the center of the specimen's length; the unsupported length can vary. In this fixture, specimens are loaded by anvils of nominally the same thickness as the specimen. The face supports extend beyond each end of the specimen to provide lateral support for the anvils.

This design was extended to air testing and the 2800°F Air Compressive Facility is shown in Figure 3.1-8. In this setup, specimens are

radiantly heated by U-shaped silicon carbide elements. The U-heaters also acts as the support for the specimen. Fibrous zirconia board provides thermal insulation. Power is supplied and controlled by a 5KVA variable transformer. The furnace and specimen are exposed to the laboratory atmosphere to provide an air environment. Temperature is monitored by an optical pyrometer. Readings are taken through an open sight port in the zirconia board. Strain measurements are made on opposite edges of the specimen. As in the tensile tests, flags are attached to the specimen by grooves in the specimen or in ceramic paint coated on the edge of the specimen. Instrumentation is basically the same as that in a tensile test.

This basic test can be put in a servo-hydraulic machine, or a creep frame for fatigue and stress rupture/creep testing.

3.1.3 Interlaminar Shear Testing to 2800°F in Air

Interlaminar shear strength is determined using the double-notch shear specimen. A typical configuration is given in Figure 3.1-9. This specimen is run in the 2800°F Air Compressive facility. From a plot of load versus time, shear strength is determined.

3.1.4 Precision Elastic Limit Testing

In materials such as ceramic and metal matrix composites, the onset of matrix cracking and the fiber/matrix interphase interaction are important. However, measuring this property can be difficult because it may involve measuring deformations on the order of microinches. Southern Research developed a precise strain measuring system that allows these measurements to be made. To measure these small deformations, the Tuckerman optical strain gage is used. A simplified schematic of the Tuckerman gage is shown in Figure 3.1-10. Basically, the gage consists of a fixed mirror and a rotating lozenge. Light emitted from an autocollimator is reflected by the lozenge to the fixed mirror and back to the autocollimator through a series of lenses. The image is projected onto a numerical scale. By tracking the movement of the image, it is possible to measure the deformation of the specimen. Deformations as small as two microinches can be measured.

To conduct a PEL evaluation, the Tuckerman gages are placed on the edges of the specimen. Alignment is checked by loading the specimen a small amount, reading the outputs of the Tuckermans, and adjusting the load train until the outputs are the same within a small percent. Once alignment is satisfactory, the specimen is loaded a predetermined amount and deformation is measured; the specimen is unloaded and return deformation is measured. This same load level is repeated three times. If yielding occurs, there will be permanent deformation upon return. The specimen is then loaded in increasing increments, repeating the load-unload sequence, until it is certain that microyielding has occurred. A plot of stress versus permanent strain will give the PEL stress. Figures 3.1-11 is a plot obtained from PEL testing of Nicalon/CAS ceramic matrix composite. This type of behavior is observed in both ceramic and metal matrix composites.

Although this test is normally run at room temperature in a closely controlled thermal environment, work has been done to exte -' the technique to 800°F. At this temperature, there is a loss in accuracy from 2-4 microstrain to 10-20 microstrain.

The Tuckerman optical strain gages are also used in dimensional stability tests for materials such as Beryllium Oxide, where very small creep strains (less than 10 microstrain) have been observed at room temperature.

3.1.5 Other Mechanical Test Techniques

When the program began, Iosipescu shear and flexural testing were within the scope of the contract. However, these methods were never brought to fruition because none of the material development efforts required these particular properties. Ideas for both were discussed and the methods are easily implementable; however, in the interest of using funds for other immediate tasks, actual test fixtures were not built.

3.1.6 Furnace for 4000°F Air Experiments

An induction heater air furnace was designed and demonstrated for operation to 4000°F. A schematic is shown in Figure 3.1-12. The objective of

this effort was to provide a laboratory furnace that could be used to heat treated and mechanically test materials to 4000°F in air. The tensile furnace could handle temperatures to 3200°F and this design would extend temperatures to 4000°F.

Three zirconia materials were found that would suscept at 3 or 4 kilohertz after being preheated to 2300°F and could then be taken to 4000°F with no other assistance other than induction heating. The heaters would occasionally blister but there was little trouble with cracking at he rates of about 4000°F/hour. For initial preheating, a double layer of graphite yarns were wrapped around the zircona and allowed to burn off at 2300°F where the direct induction of the zirconia took over. The power unit had a rating of 25 KW.

All of the insulation was consolidated zirconia fiber produced by Zircar in either flat plate from which washer could be cut and stacked or as cylinders. The material shrinks and cracks but still performs its mission and is easily replaced. Often, quartz string was wrapped around the OD to close or restrain the cracks.

Even though we had no 4000°F material to test, a few experiments were conducted with cooled pullrods in the ends where the plugs are shown in Figure 3.1-12. The pullrods were insulated to hold 1600°F. Dummy silicon carbide specimens "melted." The power to the heater was sufficient and the temperature gradient was an acceptable 100°F.

The furnace will be sufficiently rugged and flexible to handle 4000°F mechanical specimens when they become available.

3.2 Uniform Oxidation of 2-Dimensional Carbon-Carbons

Graphitic carbon composites have been proposed for use as a structural material in high temperature oxidative environments. In order to determine the relationship between mechanical properties and the degree of oxidation of a material, a uniform state of oxidation is required. Southern Research developed a test methodology to uniformly oxidize 2DCC materials and

then mechanically characterize the material as a function of extent of the oxidation. The details of the methodology have been previously released in the first of two subtask reports, SRI-MME-90-503- 6145-23 "Uniform Oxidation of Two-Dimensional Carbon/Carbon Composites: Part I - Procedural Development and Physical Characterization," submitted to the Air Force in March 1991.

From the study of two carbon-carbon materials (Vought ACC-4 and Hitco 139E), it was concluded that uniform weight loss through the thickness can be achieved in 2DCC's. Generally, oxidation begins at the surface and progresses toward the center of the specimen. Weight loss gradients were lowered by reducing the oxidation temperature. In addition, there was evidence that in the trade off between higher gas flow rates and small pressure gradients, that higher forced gas flow rates were more important in reducing these gradients. The data indicated that lower oxidation temperatures may be opening initially closed porosity, whereas higher temperatures appeared to enlarge existing open porosity. Microstructural analysis of both materials indicated that the matrix was more reactive than the reinforcing filaments or the CVD carbon.

The second subtask report SRI-MME-91-232-6145-23 entitled, "Uniform Oxidation of Two-Dimensional Carbon/Carbon Composites: Part II - Mechanical Property Degradation of Hitco 139E as a Function of Extent of Oxidation," March 1992, described property versus weight loss. Figures 3.2-1 through 3.2-3 show tensile, compressive, and interlaminar shear strength versus weight loss. Rapid loss of warp compressive and interlaminar shear strength with weight loss was observed. This is consistent with the fact that matrix is first attacked by oxidation and shear and compressive properties are greatly influenced by matrix properties. No appreciable warp tensile strength was observed until weight losses exceeded 4%. This is consistent with other fiber oxidation studies that indicated no fiber strength is lost until weight losses exceeded 5%.

3.3 Oxidation Methodology at UTRC

UTRC developed oxidative test methods to monitor mass changes and to allow identification of gaseous species evolved during thermal exposures of composites.

The simplest initial tests utilized laboratory air furnaces to perform isothermal and cyclic tests where measurements were made after defined time intervals on silicon nitride coated Hitco 2DCC. The oxidative behavior was consistent with previous data where very little oxidation was observed at 2550°F while extensive substrate oxidation and corresponding mass loss was observed at lower temperatures. The maximum oxidation rate was at 1500°F. This is explained by the fact that the coatings are microcracked, and the oxidation rate of the coated substrate is a function of the crack width and the intrinsic oxidation rate of the substrate both which vary inversely over the temperature range.

To measure continuous mass changes in oxidizing environments up to 3000°F, a state-of-the-art Cahn 1000 microbalance was coupled with a molydisilicide resistively heated furnace. The balance is interfaced with an IBM PC for continuous data logging. Computer software allows thermogravimetric data to be plotted.

To measure evolved chemical species during oxidation testing, a high temperature inductively heated enclosure was interfaced with a mass spectrometer. In this system, mass flow controllers pass controlled gas compositions into the heated enclosure where advanced composite specimens are exposed. Part of the gases leaving the hot zone are sampled with a quadropole mass analyzer, with the remaining exhaust gases being vented.

Further details of these facilities can be found in SRI interim report SRI-MME-90-393-6145 entitled "Mechanical Behavior of Composites at High Temperature," submitted to the Air Force in October 1990 and UTRC's final subcontract report R90-917601-28 entitled "High Temperature Composites Mechanical Behavior," submitted in September 1991.

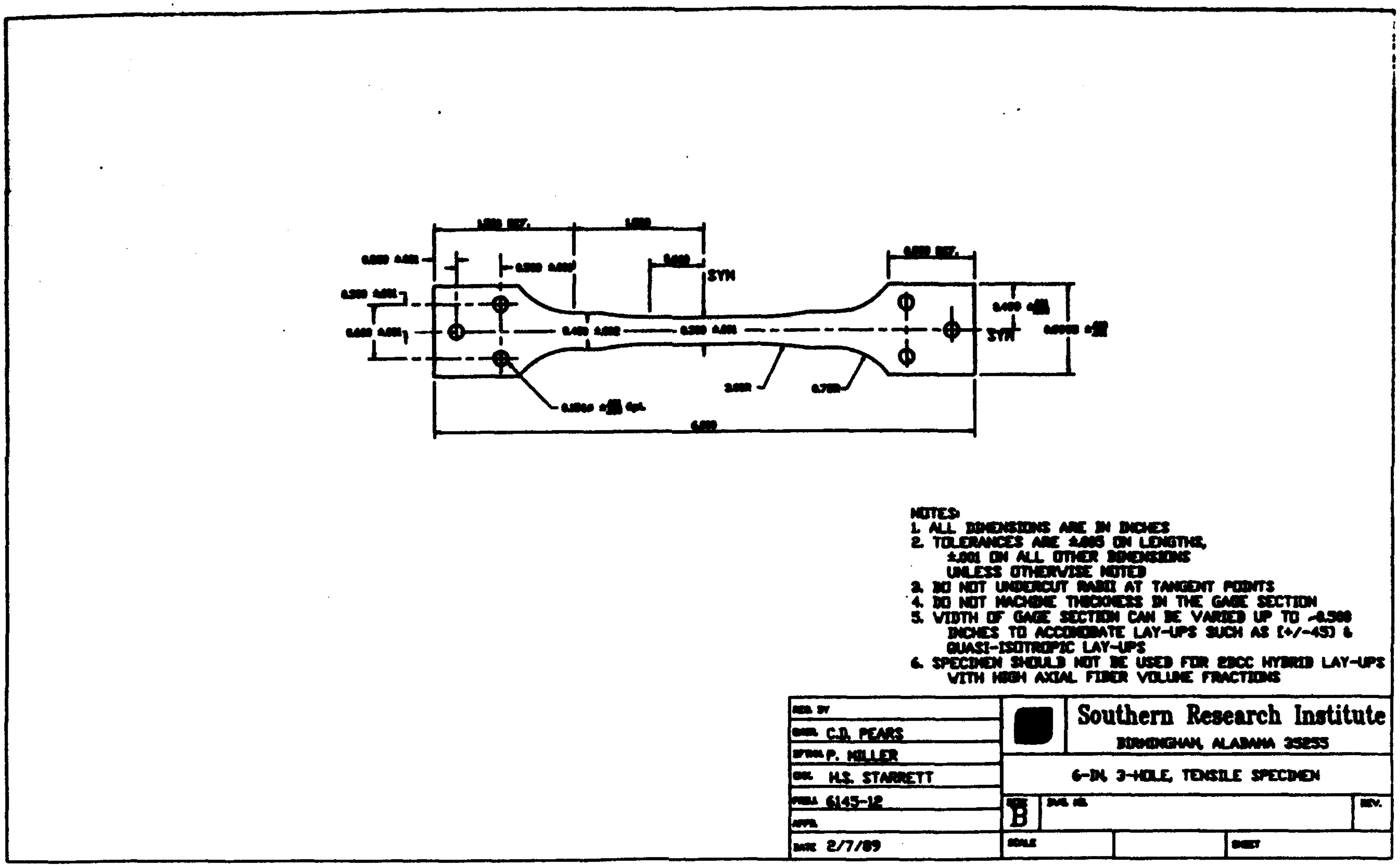


Figure 3.1-1. Typical Tensile Configuration for Ceramic Matrix Composites

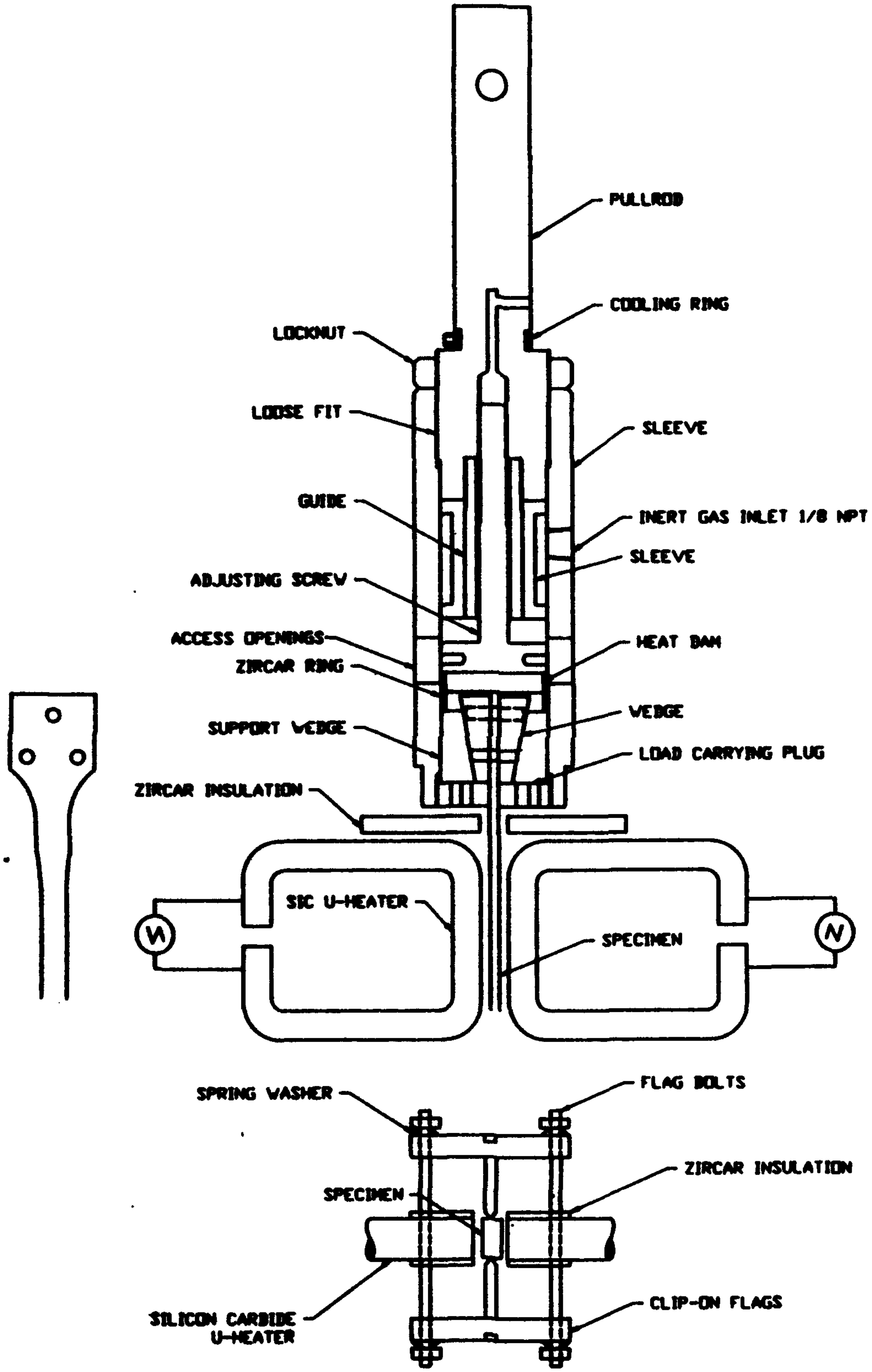


Figure 3.1-2. Schematic of the Specimen Gripping/Heating Technique

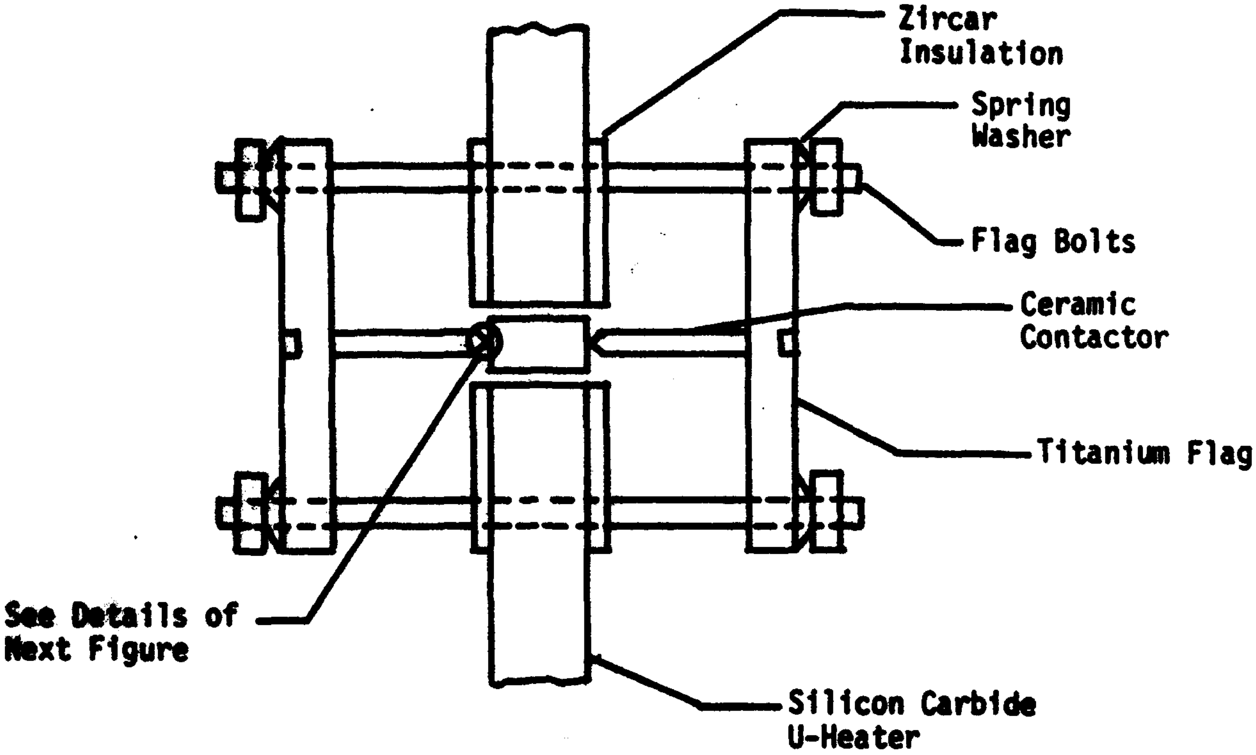


Figure 3.1-3. Schematic of Clip-on Extensometer Flags for Measuring Strain

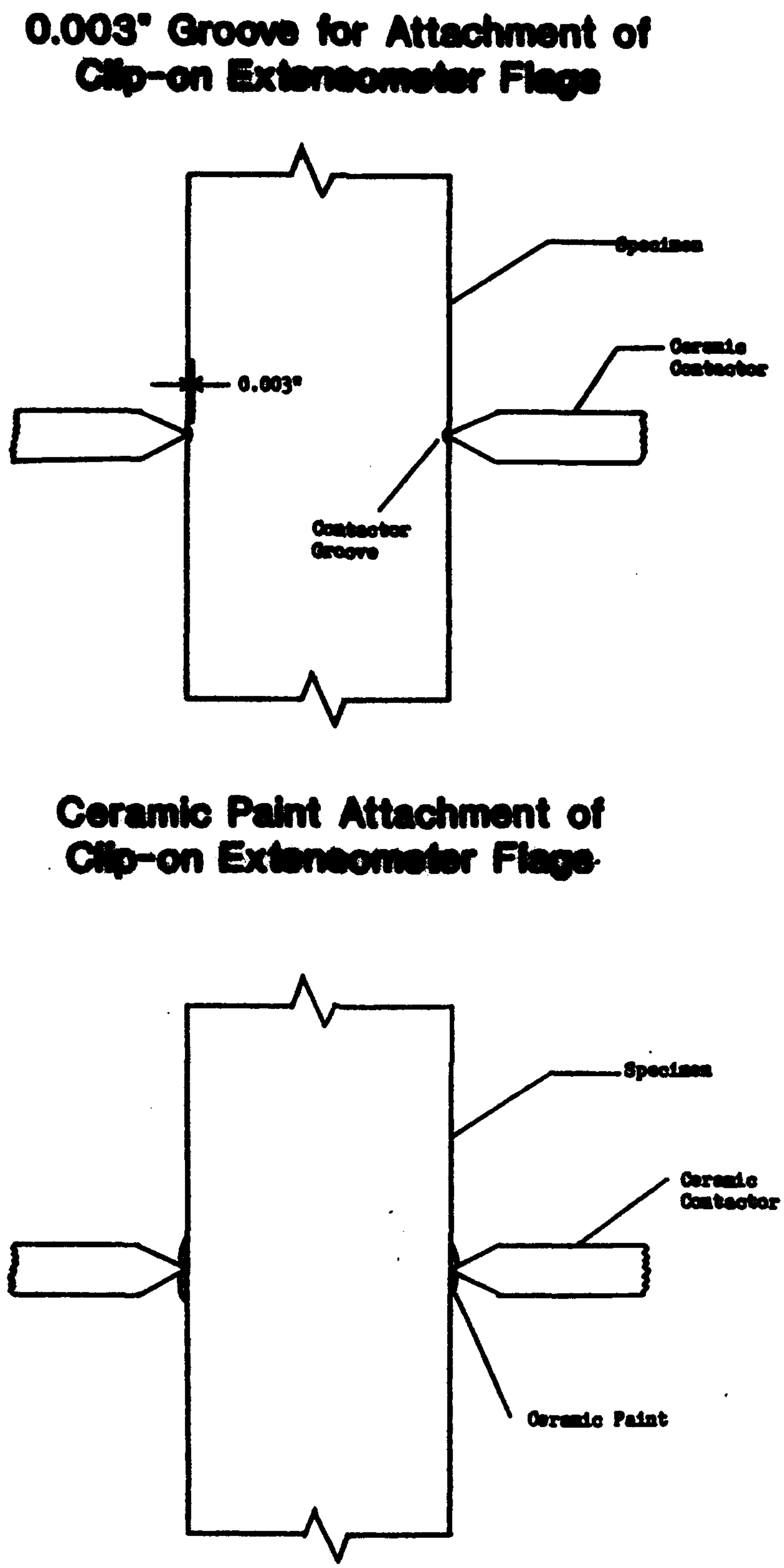


Figure 3.1-4. Attachment Techniques for Clip-on Extensometer

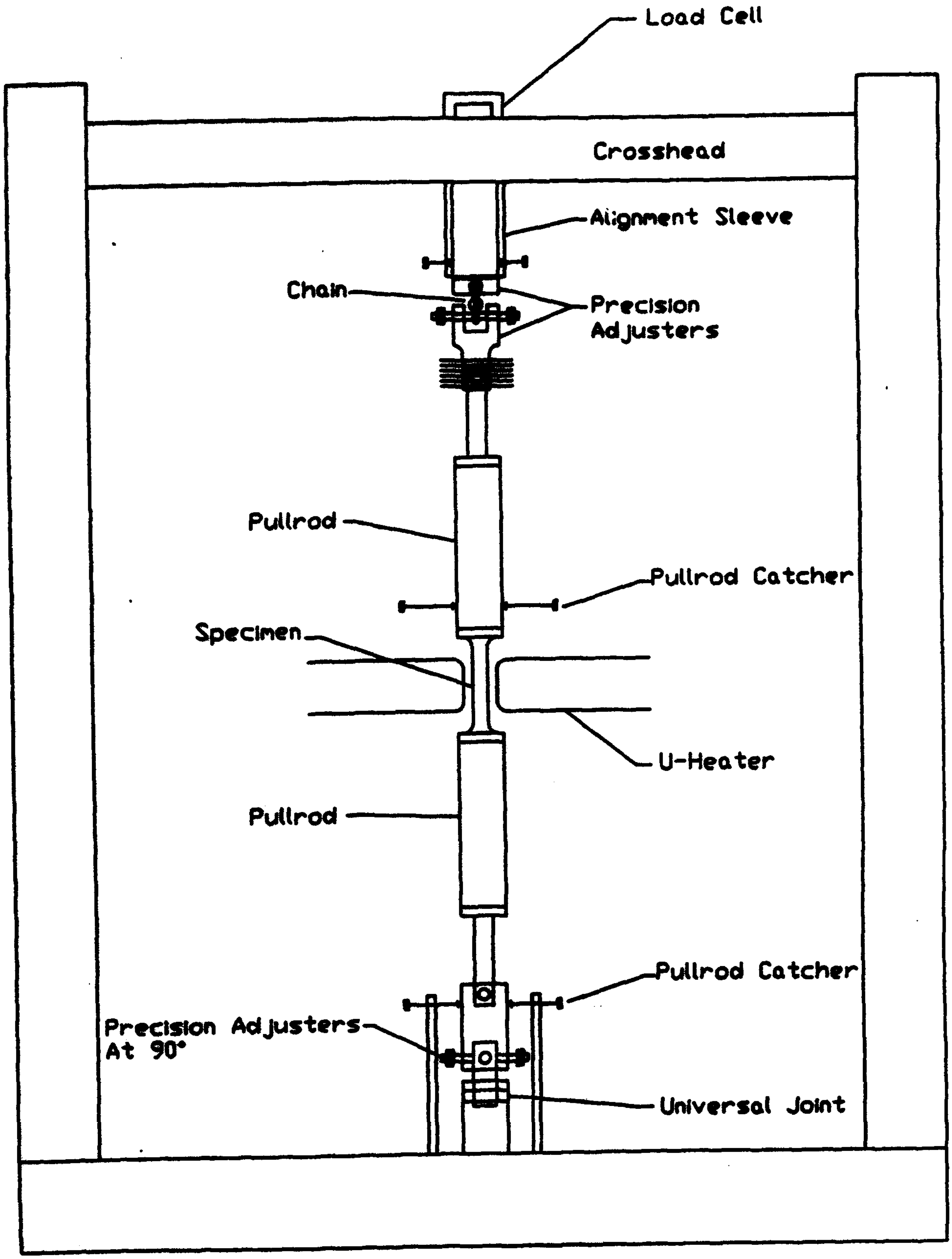


Figure 3.1-5. Schematic of Air Tensile Set-up

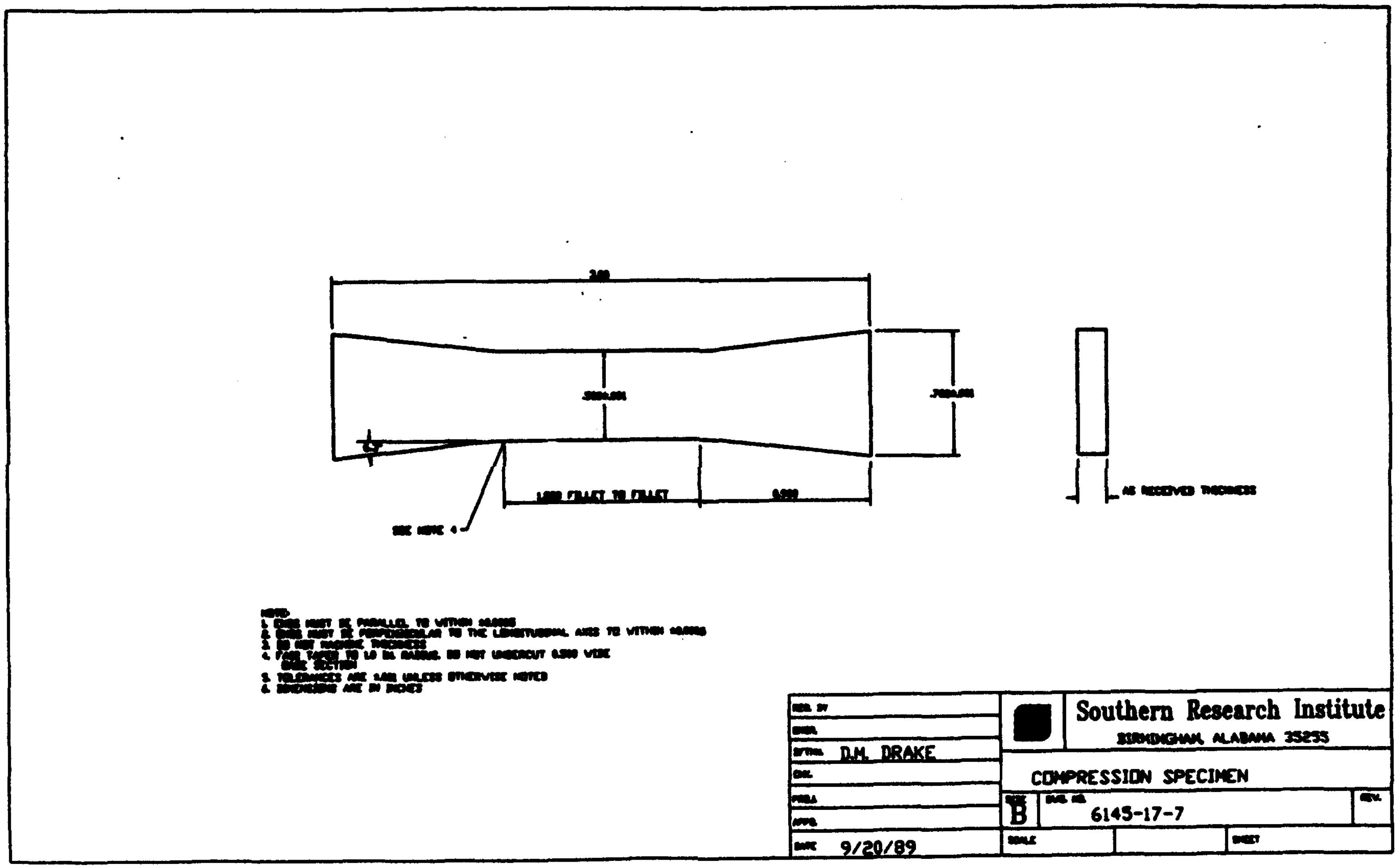


Figure 3.1-6. Typical Compressive Configuration for CMC's and CC's

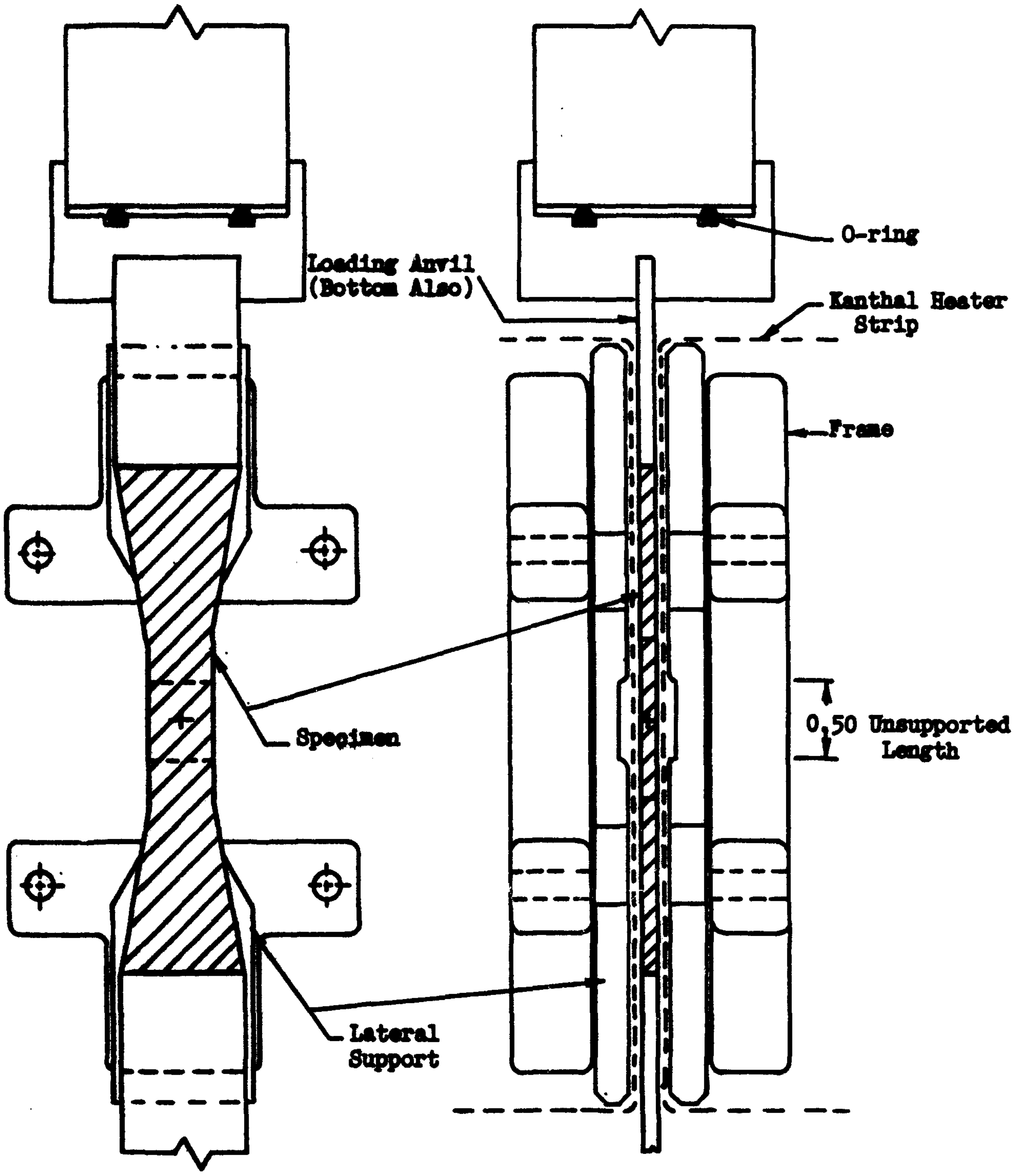


Figure 3.1-7. Schematic of Southern's Controlled Face Supported (CFS) Compressive Fixture

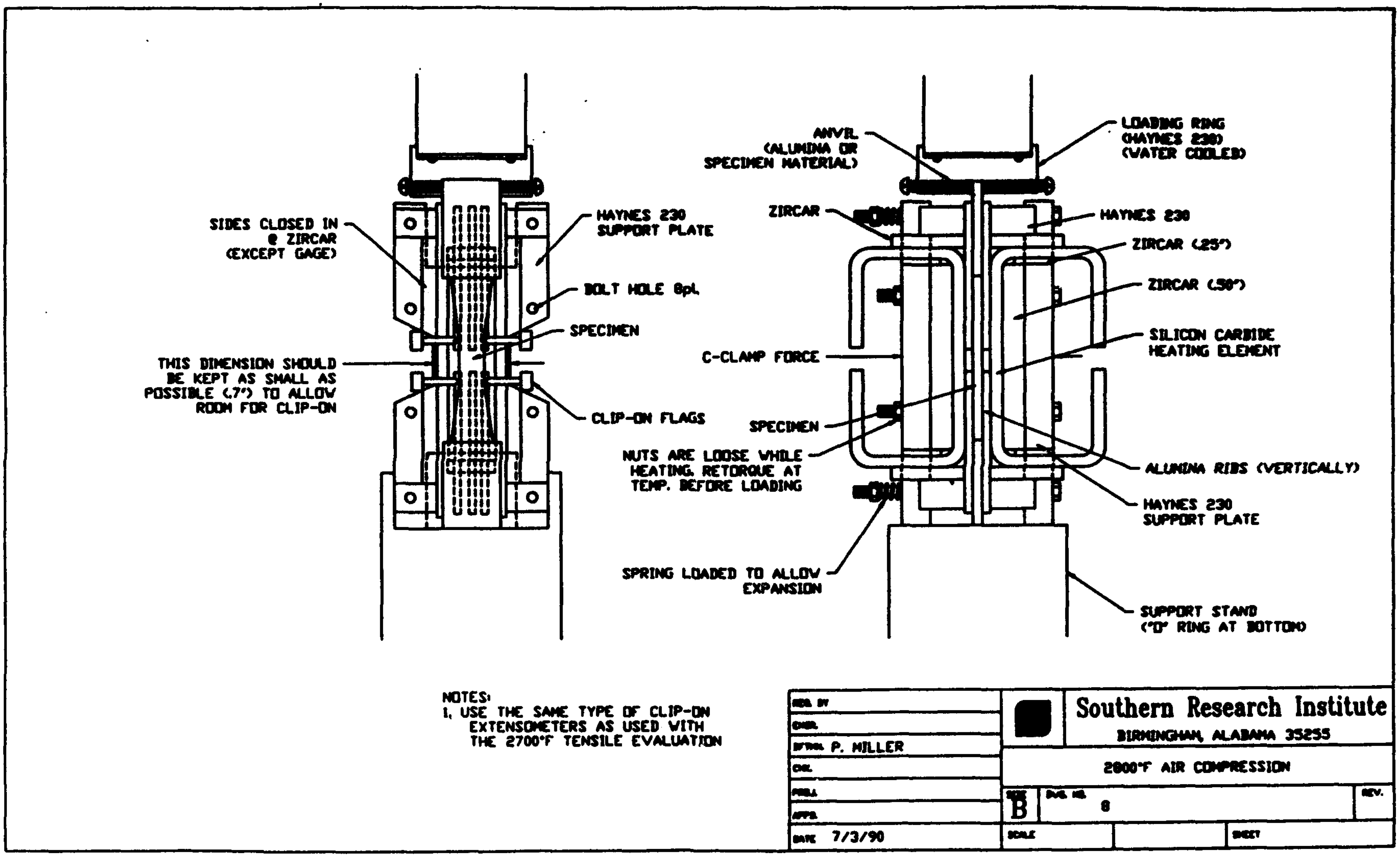
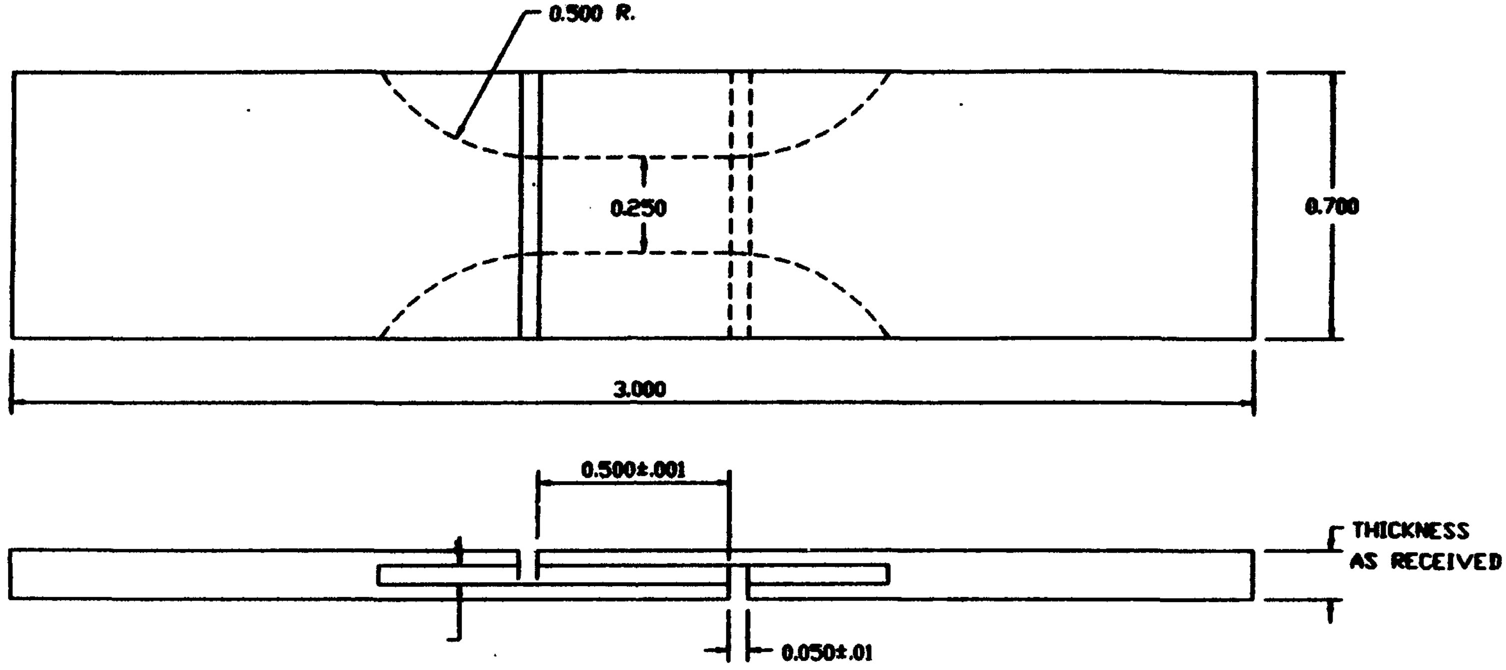


Figure 3.1-8. Schematic of the 2800°F Air Compressive Facility



NOTES:
1. ENDS MUST BE PARALLEL TO WITHIN ±.0005 IN.
2. ENDS MUST BE PERPENDICULAR TO LONGITUDINAL AXIS TO WITHIN ±.0005 IN.
3. DO NOT UNDERCUT 0.500 IN. GAGE SECTION
4. TOLERANCES ARE ±.005 UNLESS OTHEERWISE NOTED
5. ALL DIMENSIONS ARE IN INCHES

Figure 3.1-9. Typical Interlaminar Shear Configuration

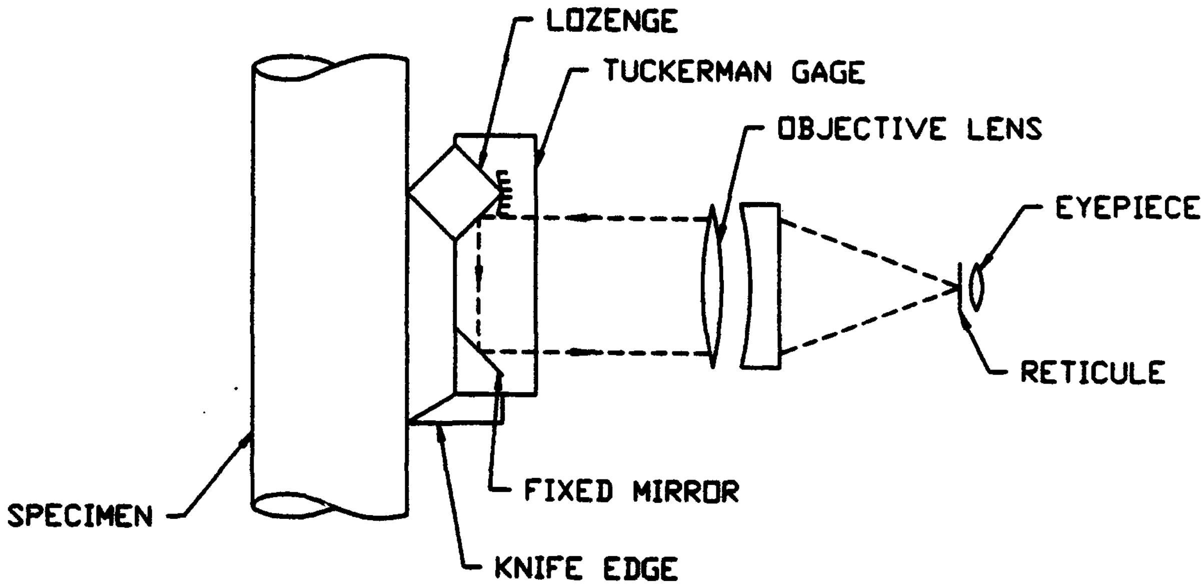


Figure 3.1-10. Schematic of the Tuckerman Optical Strain Gage

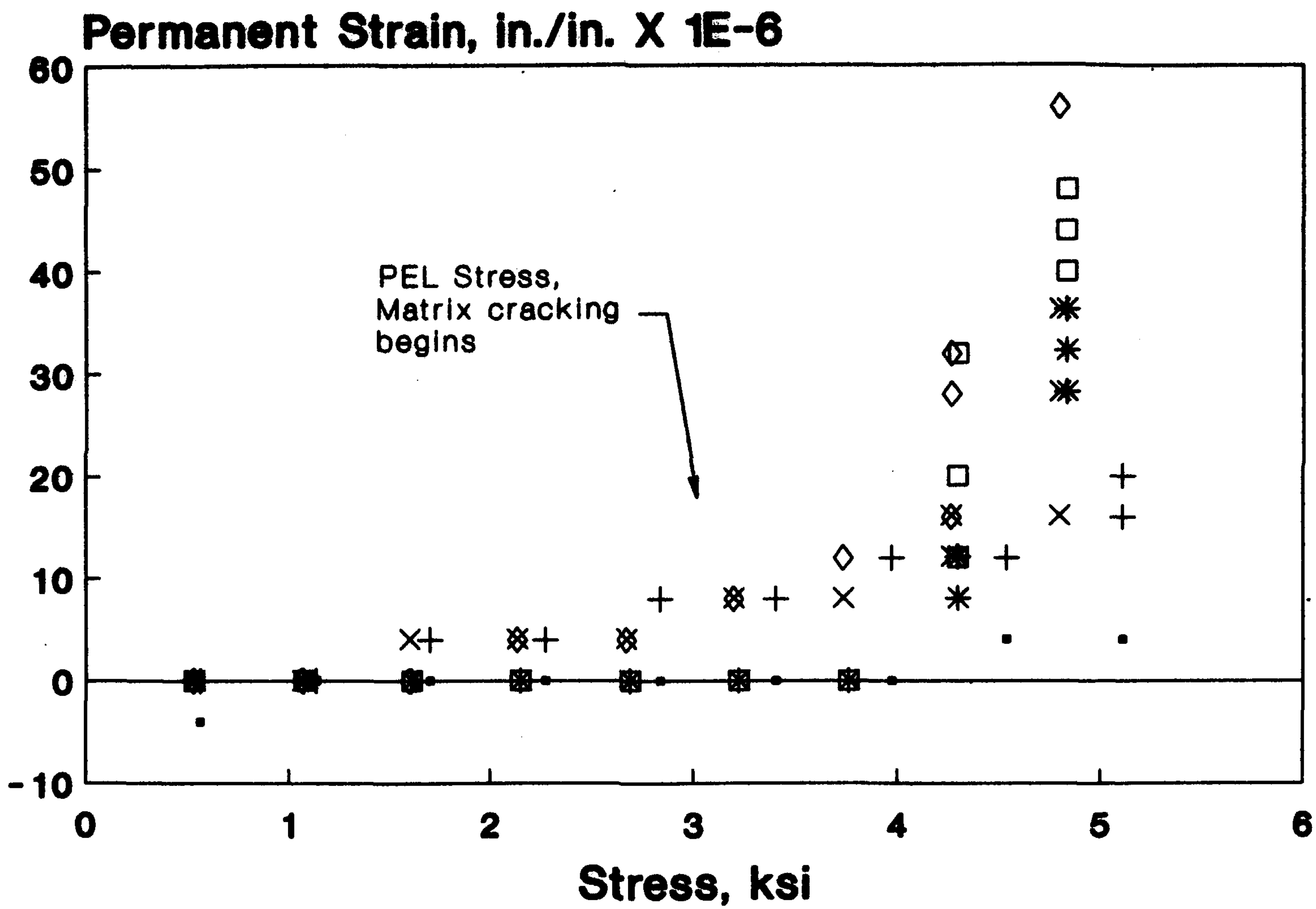


Figure 3.1-11. Permanent Strain versus Stress for Nicalon/CAS CMC

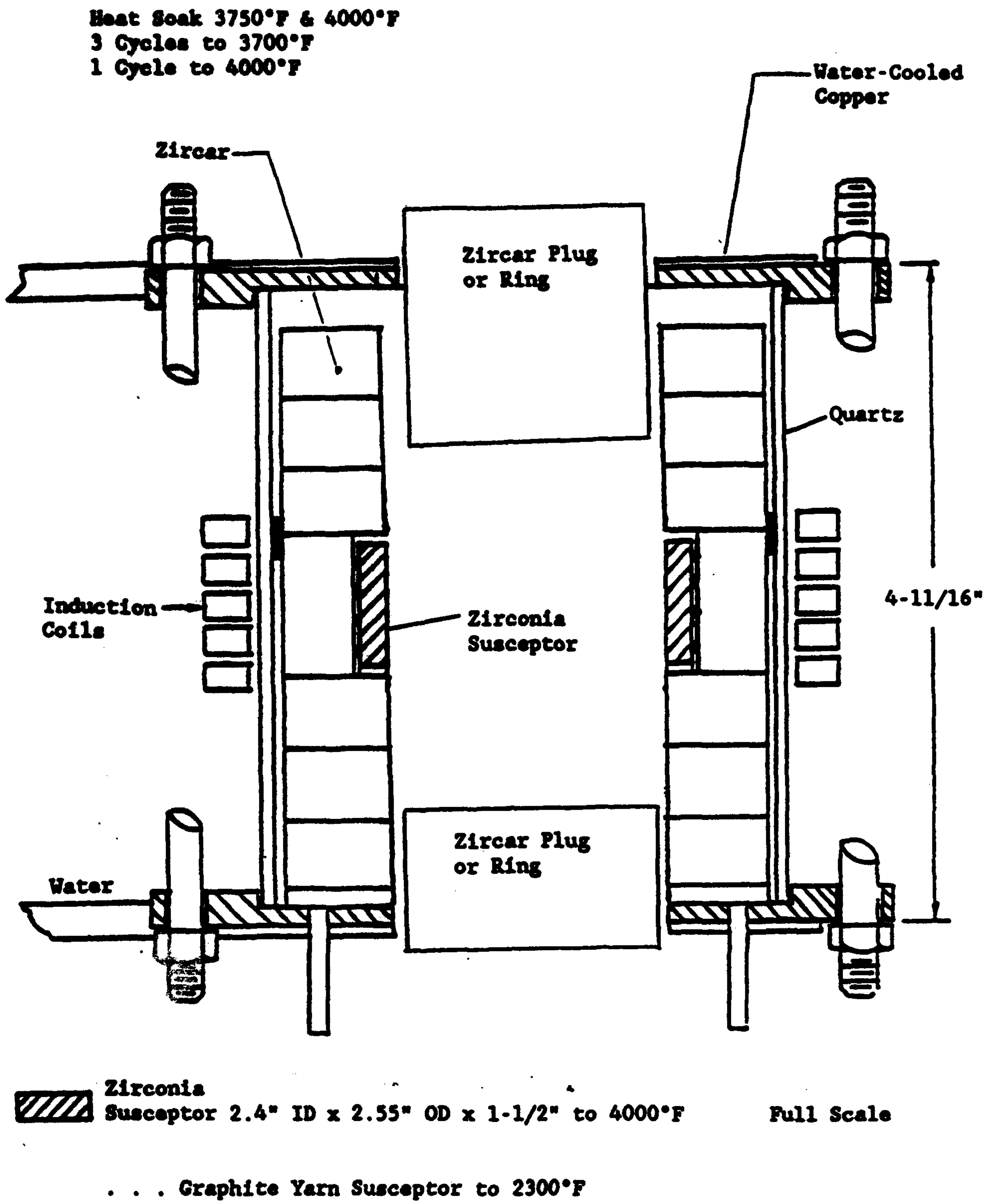


Figure 3.1-12. Schematic of the 4000°F Air, Puck Furnace

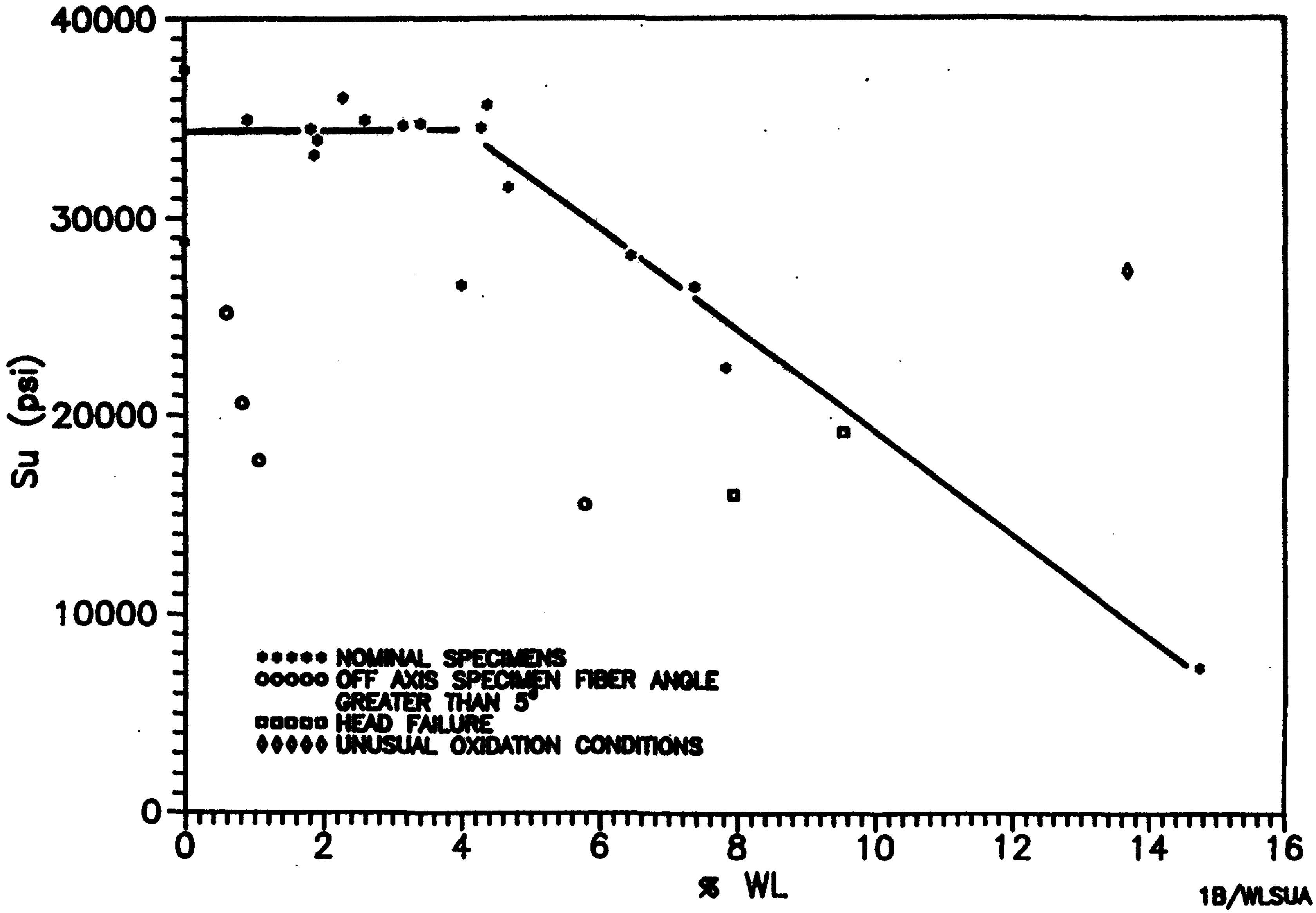


Figure 3.2-1. Warp Tensile Strength versus Weight Loss for Hitco 139E

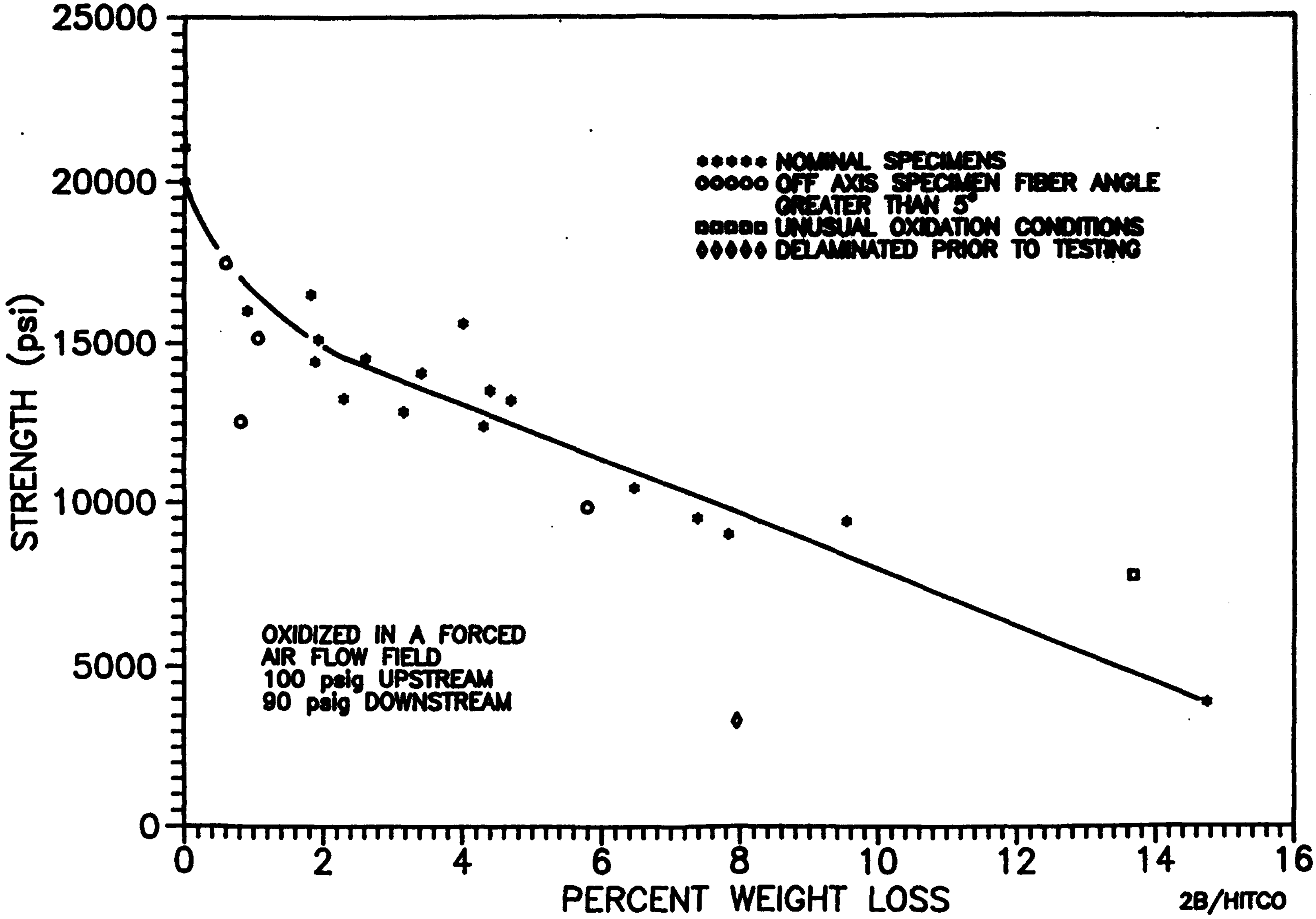


Figure 3.2-2. Warp Compressive Strength versus Weight Loss for Hitco 139E

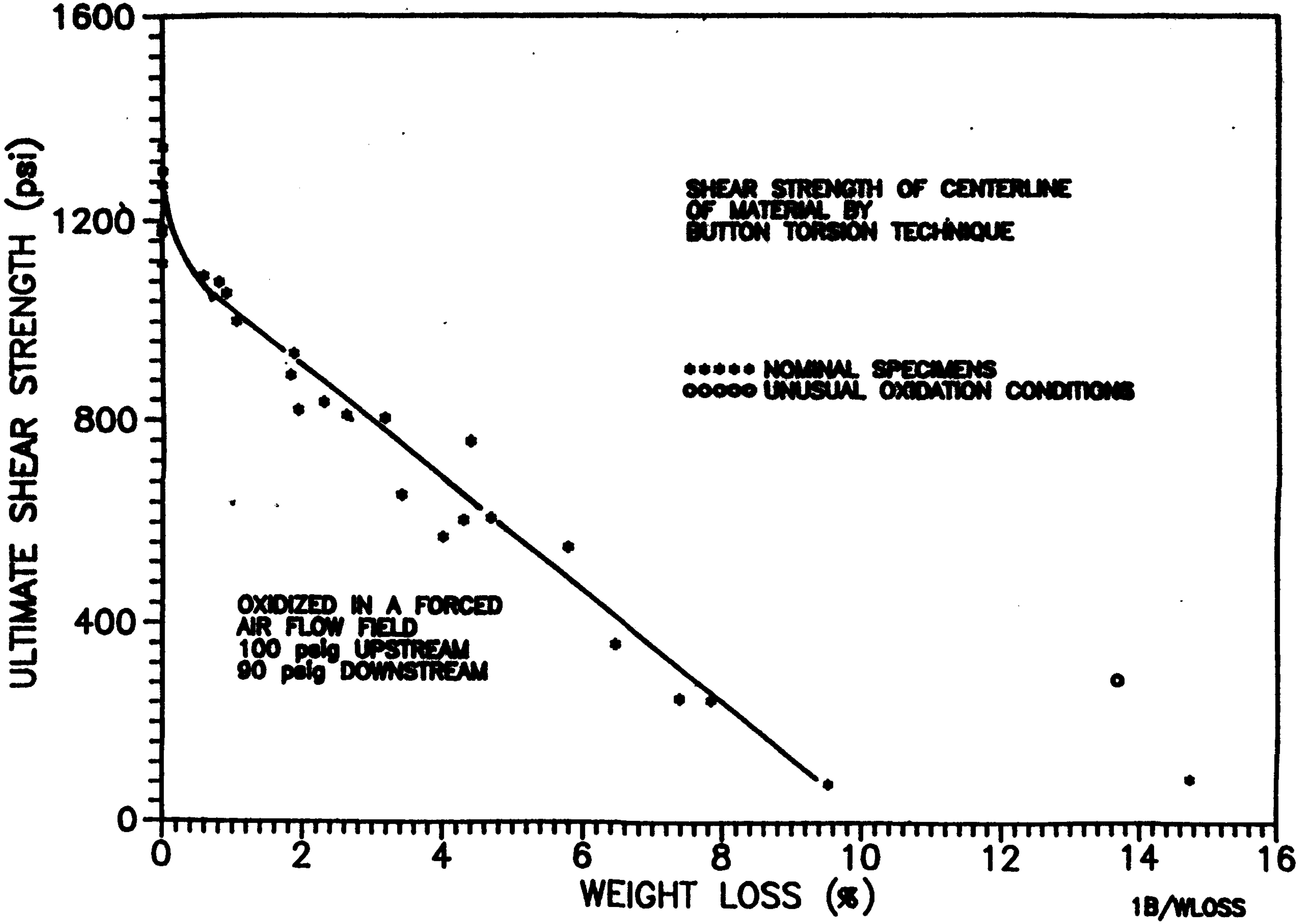


Figure 3.2-3. Interlaminar Shear Strength versus Weight Loss for Hitco 139E

4.0 MODELING AND ANALYSIS

4.1 Modeling Efforts at MSC

The two major thrusts by MSC were to examine the four-point flexure test as a potential material development tool for high temperature composites and analysis of the nonlinear tensile stress-strain response of ceramic matrix composites.

The analysis of the four point flexure test was accomplished by developing and exercising analytical models that represented realistic tensile, compressive, and shear properties of typical high temperature composites. It was found that the models could accurately predict the flexural behavior of the materials if the tensile, compressive, and shear properties were known.

The analysis of 2DCC flexural response showed that the material behavior determined from the flexural specimen was in relatively good agreement with measured tensile and compressive properties. The modulus of a 2DCC could be reasonably determined from the flexural data as long as the beam aspect ratio was 20:1 or greater. The strength of the 2DCC determined from flexure data was accurate because the material properties were relatively linear in tension and compression.

The study of 2D Graphite/SiC showed flexural response can be extremely misleading. The flexural data resulted in good modulus measurements; however, flexure data significantly overestimated the tensile strength and underestimated the compressive strength because of the nonlinear nature of the material stress-strain curves.

These results implied that flexural data may be misleading. In some cases, flexural testing may be appropriate but in other cases, the tests may lead to incorrect assessments. Unfortunately, the only method for determining the value of flexural data is to examine tensile, compressive, and shear stress-strain responses from the same material. If this data is already available, the flexural data is probably no longer needed. This leads to the

conclusion that a good tensile or compressive test is more valuable than a flexure test.

Progressive damage analyses were performed to examine the nonlinear tensile behavior of unidirectional and cross-ply Nicalon/CAS composite laminates. The analytical results were compared to test data measured by Southern Research. The results showed that there will be very intensive matrix cracking in the material once the material is stressed beyond its first ply failure stress. Consequently, if a ceramic structure is being designed to have little or no internal cracking so that it can survive oxidizing environments, the allowable stress should not approach anywhere near the first ply failure stress.

Details of the flexure and tensile analyses can be found in SRI interim report SRI-MME-90-393-6145 and MSC's final subcontract report TFR 3114/4009 entitled "Mechanical Behavior of High Temperature Composites: Material Modeling," submitted in October, 1991.

4.2 Modeling Efforts at VPI

VPI addressed the micromechanics of short-fiber composites, microthermal stress analysis with local property gradients, and predictive modeling of remaining strength and life. The details of their analysis can be found in VPI final subcontract report entitled "Performance Modeling of Ceramic Composite Materials," submitted April 1990.

5.0 MATERIAL CHARACTERIZATION

5.1 Physical, Mechanical, and Thermal Evaluation

One of the main goals of the program was to evaluate state-of-the-art ceramic composites and oxidation protected carbon-carbon composites up to the maximum potential use temperature using available test and evaluation techniques. Southern Research assigned as specific subtask number and budget for the material to be evaluated. Each material was mechanically and/or thermally characterized using the technology available, including the new air testing capabilities developed. Examples of data generated from these efforts are given in Appendix A.

5.2 Microstructural Evaluation at UTRC

UTRC performed microstructural evaluation of five different composites materials:

1) Arco Al_2O_3 Whisker/SiC Matrix Composite
2) Corning Nicalon Fiber Reinforced Calcium Alumino Silicate CMC
3) Kaiser Nextel 480 Fabric Reinforced SiC
4) T-40R Fiber Reinforced 2DCC
5) Rohr T-300 Inhibited 2DCC

The analyses were implemented to correlate with mechanical testing at Southern Research and to determine any distinguishing characteristics on both the macro- and micro-scales. Metallographic and X-ray diffraction analyses were utilized to define coarse microstructural features, while Auger analysis, analytical TEM, and SEM were used to study the fiber/matrix interface characteristics. The details of the evaluations are given in UTRC's final subcontract report R90-917601-28 entitled "High Temperature Composites Mechanical Behavior," submitted in September 1991.

5.3 Fatigue Testing at GE

GE assisted Southern Research in the development of elevated

temperature test techniques for generating creep rupture and fatigue data on composite materials. Fatigue data was generated on Corning Nicalon/CAS and Chromalloy RT-42 coated, Rohr T-300 inhibited 2DCC composites.

For the Nicalon/CAS CMC, the following conclusions were made:

1) The room temperature fatigue behavior exhibited a log-linear response as a function of maximum stress. The elevated temperature (1500°F and 1800°F) fatigue performance dropped rapidly from the static tensile value of 20 ksi and then quickly leveled off at 11 ksi.

2) All specimens exhibited significant stiffness degradations during the course of fatigue testing.

3) Post-test evaluation of fatigue specimens indicated substantial microcracking and fiber bridging.

For the 2DCC specimens, the following conclusions were drawn:

1) 2DCC exhibits a very flat fatigue response at room temperature with residual strengths of the runout specimens greater than initial static strengths.

2) 1200°F inert fatigue results exhibit a flat fatigue response. However, 1200°F and 2000°F in an oxidizing environment indicate a flat response for about 8 hours and then fatigue performance drops quickly.

3) 1200°F specimens exhibited a lower performance and significantly more oxidation than the 2000°F specimens which indicates intermediate temperatures may be more detrimental to performance than higher temperatures.

These data were consistent with fatigue data generated on the same materials by Southern Research. Details of these evaluations can be found in Southern's subtask report SRI-MME-90-780-6145-21, "Room and Elevated Temperature Mechanical and Thermal Properties of Corning Nicalon/CAS," July 1990, and in GE's

final subcontract report R93AEB131 entitled "High Temperature Composite Mechanical Behavior," February 1993.

5.4 Failure Mechanisms of Oxidation Protected 2DCC

The objective of the effort was to evaluate failure mechanisms of oxidation protected carbon-carbon. Specimens were to be conditioned using the AFWAL 2550/1200°F oxidation cycle and removed at different cyclic intervals. Tests were then to be made to provide property degradation as a function of weight loss (oxidation). Later results from GE's ORCCID program showed that the 2550/1200°F AFWAL cycle was to severe so the temperatures were lowered to 2200/1200°F and 2000/1200°F.

The initial material to be evaluated was a T-40R fiber reinforced 2DCC. Screening tests on the virgin material revealed poor tensile properties. Analysis by UTRC and Rohr showed that the heat treat temperature exceeded 3000°F and the fiber was damaged.

The T-40R material was replaced by a T-300 fiber reinforced 2DCC. Tests on virgin material showed good tensile properties and coupons were removed from supplied panels, nondestructively characterized, and then sent to Chromalloy Research and Technology for application of the RT-42 SiC coating. Specimens were returned to SRI and then sent to GE for the oxidation cycling.

Initially two sets of specimens were cycled at peak temperatures of 2200°F and 2000°F. Nearly all of the specimens exhibited significant weight loss after the first cycle, and several developed large pin-holes which lead to highly localized oxidation. Subsequent discussions with Rohr and Chromalloy revealed deviations in the coating process due to gas leaks during application and other equipment problems. In addition, Chromalloy noted some damage associated with grit blast rework operations performed by Rohr. Rohr replaced the defective batch of specimens.

The two sets of replacement coupons were sent to GE and cycled to peak temperatures of 2200°F and 2000°F. The weight changes are summarized in Figures 5.4-1 and 5.4-2. These specimens exhibited little weight change as a

result of thermal cycling. Because the contract was nearing end and funding was low, further cycling was halted and the specimens were returned to SRI. No residual mechanical property tests were performed. The specimens remain at Southern Research in the event a future program has sufficient funds to continue the testing.

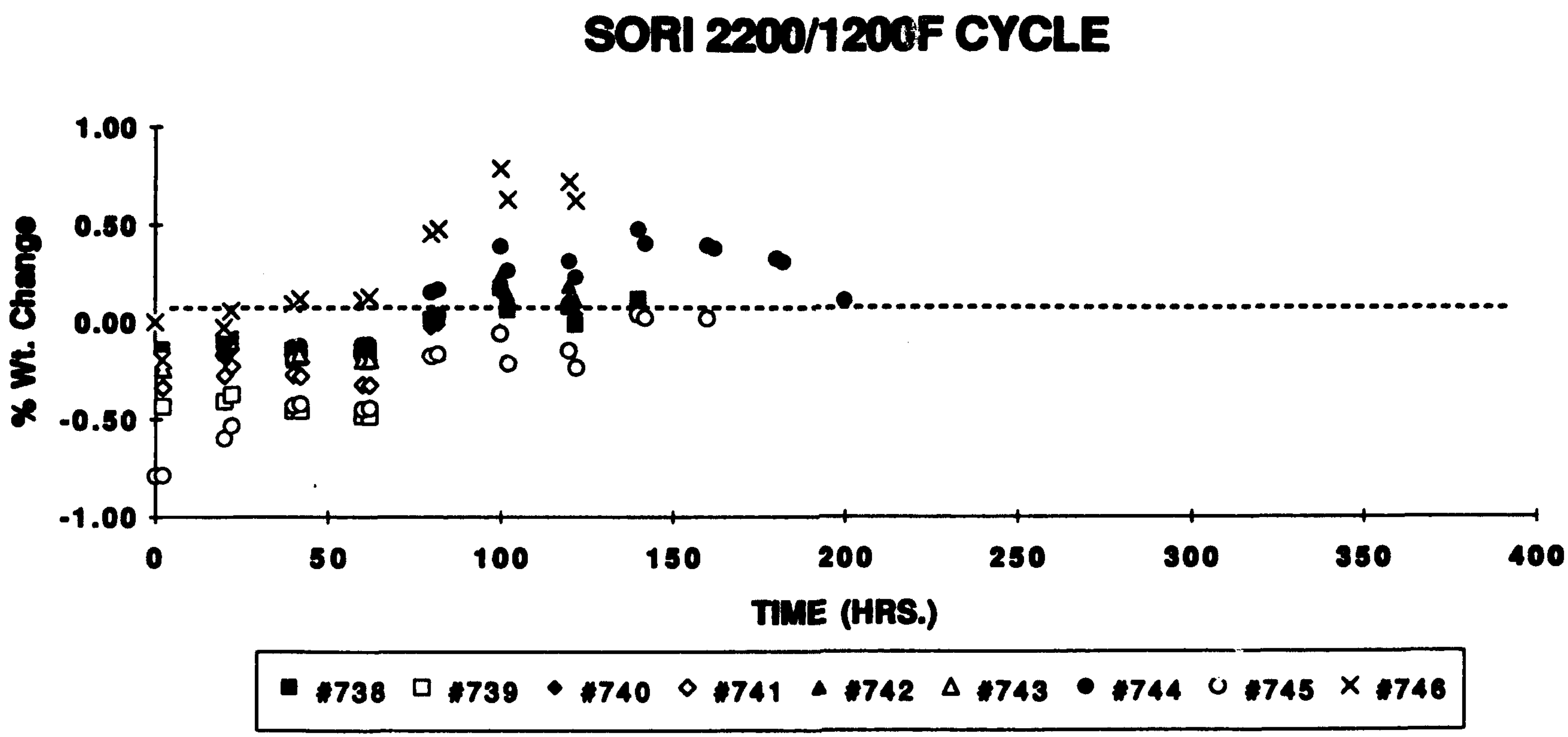


Figure 5.4-1. Percent Weight Loss versus Time for 2200°F/1200°F AFWAL Cycle

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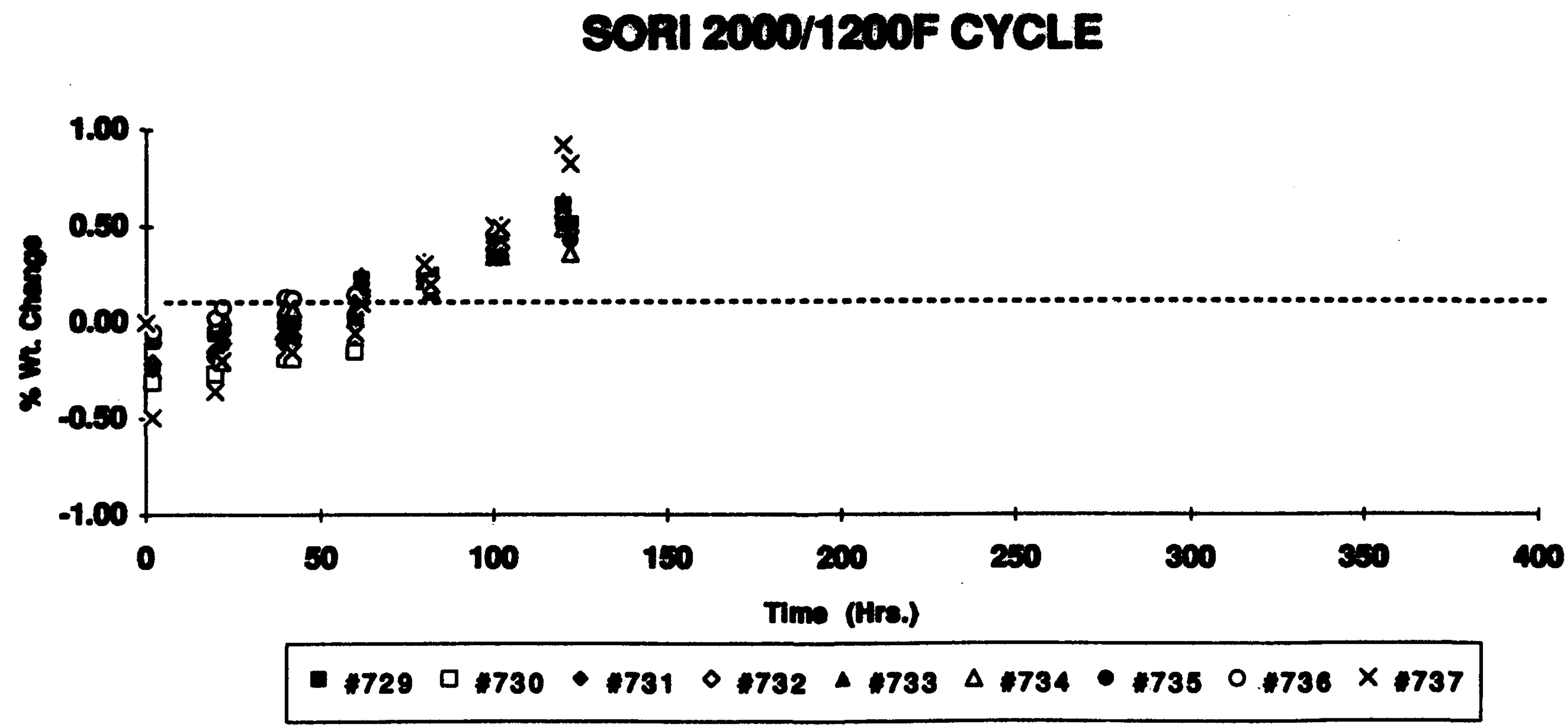

Figure 5.4-2. Percent Weight Loss versus Time for 2000°F/1200°F AFWAL Cycle

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6.0 SUMMARY

The objective of this contract was to develop an understanding of ceramic composites and oxidation-protected carbon-carbon composites, and to provide guidance for future material development of composites by the evaluation of these materials from 70°F to 4000°F in inert and oxidizing atmospheres. To accomplish this task, efforts included evaluating state-of-the-art ceramics and carbon-carbons up to the maximum use temperature, utilizing mechanics models and theories to assess the materials, improving and developing test methodologies for these materials, and developing air and high temperature heating techniques in oxidizing environments.

Although no material was developed that could withstand 4000°F environments, all other objectives were met. The results of these efforts have been released in the form of subtask reports or presentations. The following is a list of these reports/presentations:

Reports

1) Southern Research Report SRI-MME-90-393-6145, "Mechanical Behavior of Composites at High Temperature," October 1990.

2) Southern Research Report SRI-MME-90-503-6145-23, "Uniform Oxidation of Two Dimensional Carbon-Carbon Composites: Part I - Procedural Development and Physical Characterization," March 1991.

3) Southern Research Report SRI-MME-91-232-6145-23, "Uniform Oxidation of Two Dimensional Carbon-Carbon Composites: Part II - Mechanical Property Degradation of Hitco 139E as a Function of Oxidation," March 1992.

4) United Technologies Research Center Report R90-917601-28, "High Temperature Composites Mechanical Behavior," September 1991.

5) Materials Sciences Corporation Report TRF 3114/4009, "Mechanical Behavior of High Temperature Composites: Material Modeling," October 1991.

6) Virginia Polytechnic Institute Report "Performance Modeling of Ceramic Composite Materials," October 1991.

7) Southern Research Report SRI-MME-90-16-6145-17-I-F, "Physical, Mechanical and Thermal Properties of Kaiser Nextel Ceramic Composites With and Without Textron Monofilament at Room and Elevated Temperatures," May 1990. WRDC-TR-90-4133

8) Southern Research Report SRI-MME-89-1025-6145-19, "Physical, Mechanical and Thermal Properties of a Silicon Carbide Whiskers/Alumina Composite at Room and Elevated Temperatures," October 1989.

9) Southern Research Report SRI-MME-90-184-6145-20, "Physical, Mechanical and Thermal Properties of ASPC Uninhibited and Inhibited 2D Carbon-Carbon Composites at Room and Elevated Temperatures," March 1990. WRDC-90-4031

10) Southern Research Report SRI-MME-90-780-6145-21, "Room and Elevated Temperature Mechanical and Thermal Properties of Corning Nicalon/CAS," July 1990. WRDC-TR-4131

11) Southern Research Report SRI-MMER-90-751-6145-27, "Physical, Mechanical and Thermal Properties of Two RCI Graphite/Silicon Carbide 2D Composite Materials at Room and Elevated Temperature," August 1990. WRDC-TR-90-4132

12) Southern Research Report SRI-MME-89-882-6145-29, "Mechanical and Thermal Properties of a PWA Nextel 312/SiO_2 Panel," September 1989.

13) Southern Research Report SRI-MME-90-722-6145-36, "Physical, Mechanical, and Thermal Properties of the ASPC 502 and 503 Uninhibited and Inhibited 2D Carbon-Carbon Composites at Room and Elevated Temperatures," May 1991. WL-TR-91-4116

14) General Electric Report R93AEB131, "High Temperature Composite Mechanical Behavior," February 1993.

Presentations

1) SRI Presentation at the Air Force Carbon-Carbon Program Review, "Failure Mechanisms for Oxidation Resistant CC's," Dayton, OH, October 1991.

2) SRI Presentation at the JANNAF Rocket Nozzle Technology Subcommittee, "Uniform Oxidation of Two-Dimensional Carbon-Carbons," Silver Spring, Maryland, October 1989.

3) SRI Presentation at the 20th Biennial Conference on Carbon, "A Material Model for the Oxidation of a Chemical Vapor Densified Two-Dimension Carbon-Carbon," Santa Barbara, CA, June 1991.

4) MSC Presentation at the TMS Symposium on High Performance Composites, "Nonlinear Flexure Behavior of High Temperature Composites," Morristown, NJ, June 1990.

5) MSC Presentation at the 14th Annual Conference on Composites and Advanced Ceramics, "Flexure Tests as Material Development Tools for High Temperature Composites," Cocoa Beach, FL, January 1990.

6) MSC Presentation at the 15th Annual Conference on Composite Materials and Structures, "Analysis of Nonlinear Behavior of Nicalon/CAS Composites," Cocoa Beach, FL, January 1991.

Future efforts in these areas should include:

1) increasing the maximum use temperature of ceramic composites to >3000°F,

2) extending life of carbon-carbon composites with better coating systems,

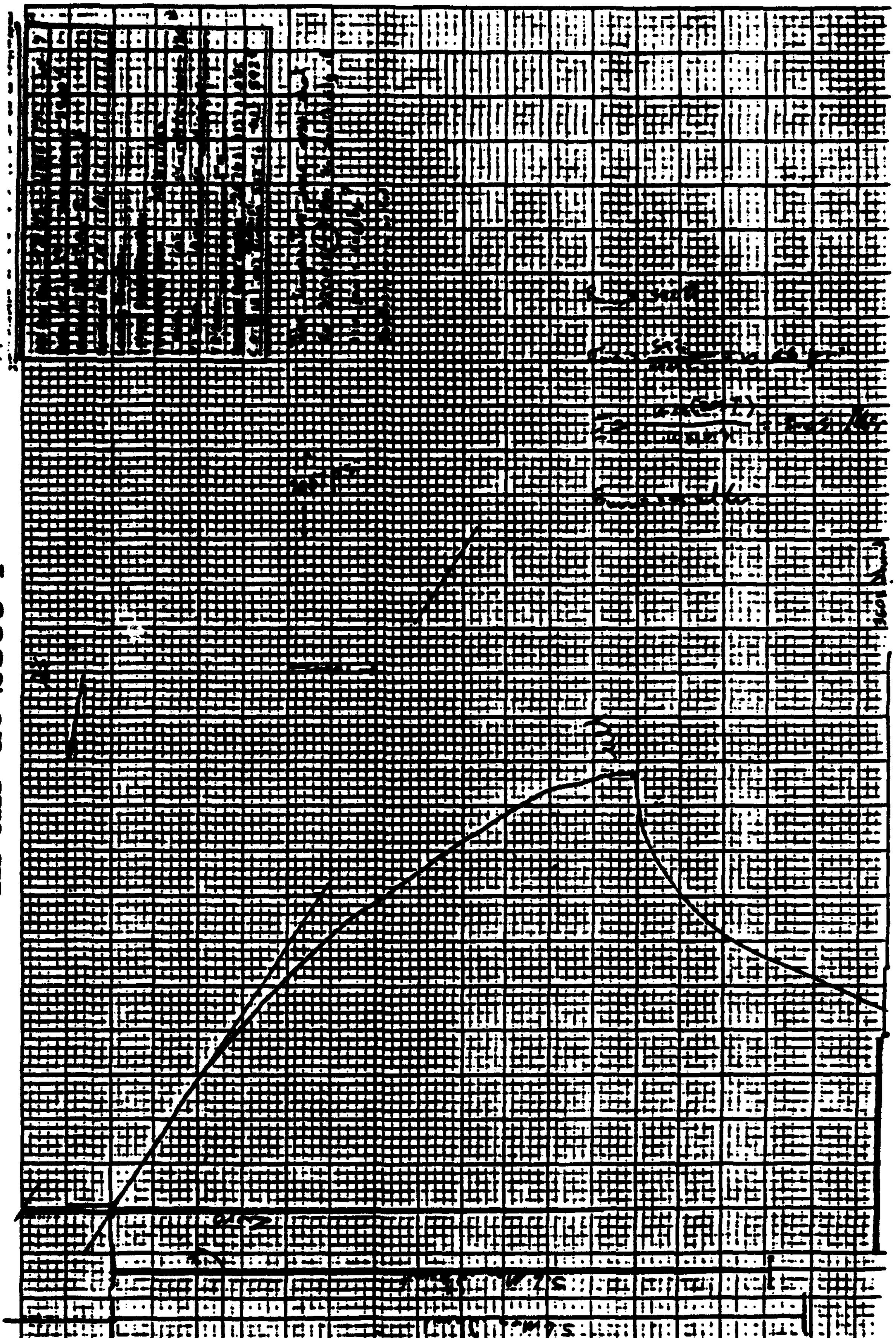
3) application of advanced models in analyzing new material development efforts,

4) extension of mechanical tests to 4000°F in oxidizing environments.

APPENDIX A

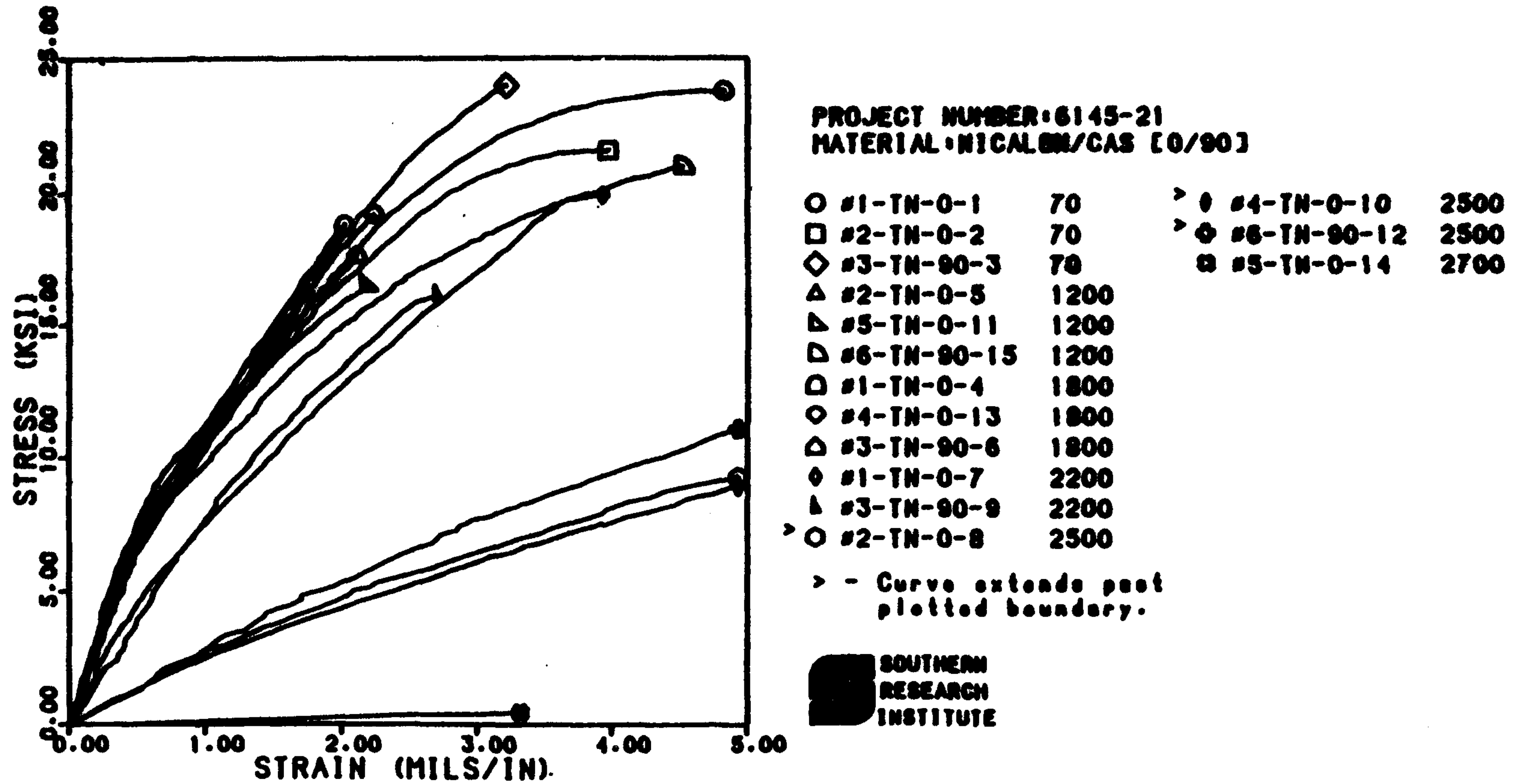
TYPICAL DATA GENERATED BY SOUTHERN RESEARCH INSTITUTE

Tensile Stress–Strain Curve in Air at 2800 F

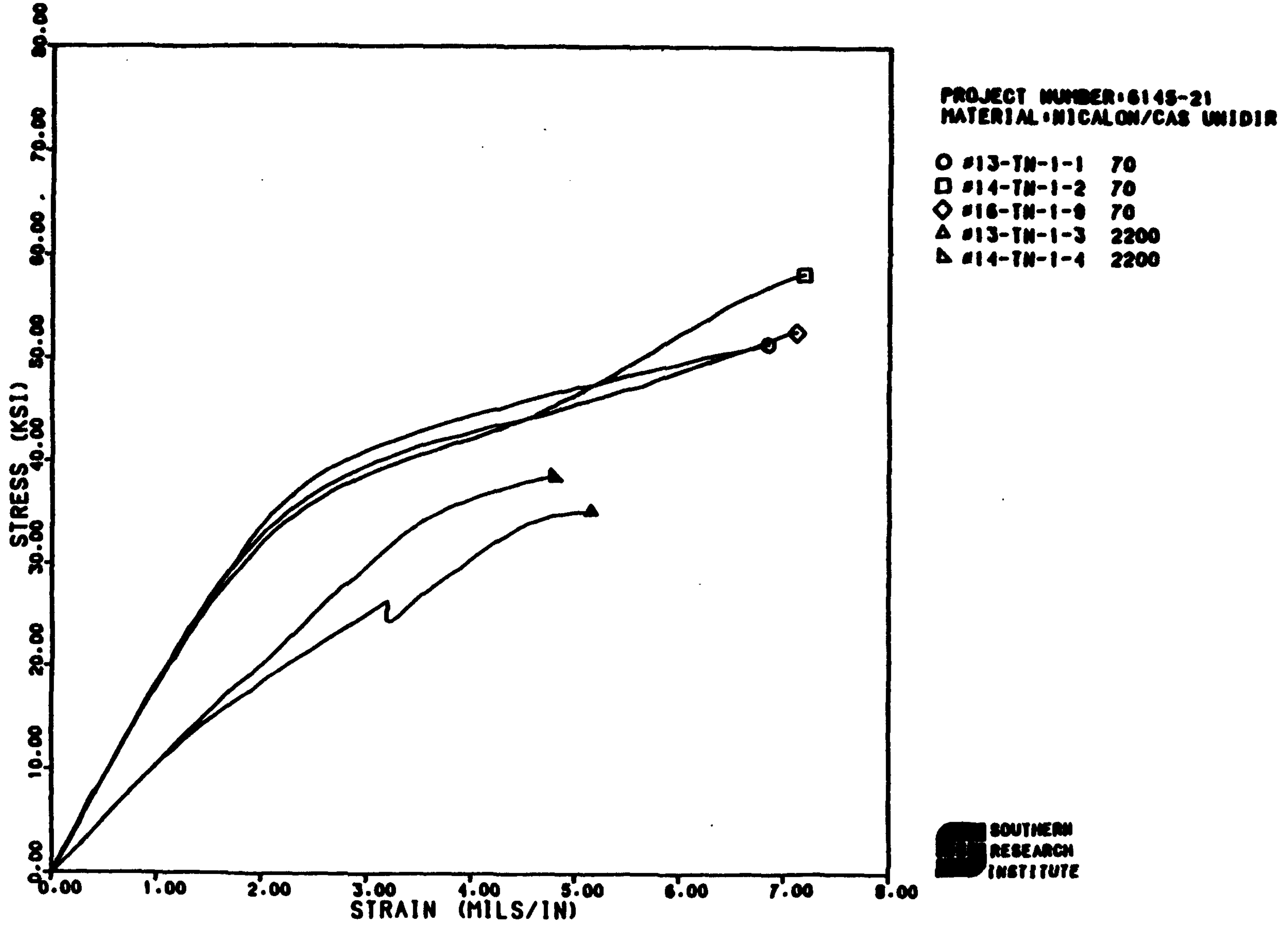


9800
2.96
.0057

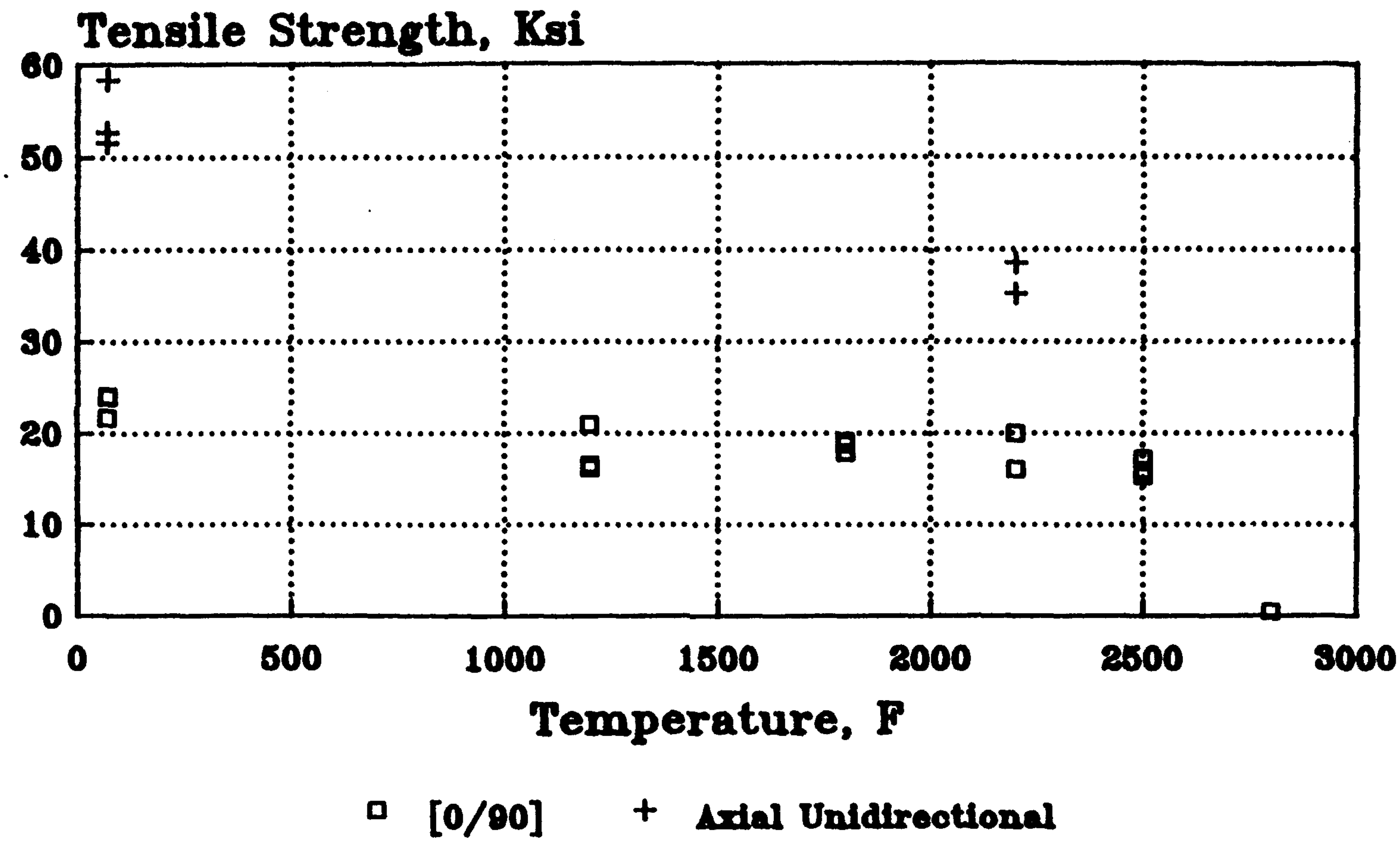
Tensile Stress–Strain Response of [0/90] Nicalon/CAS



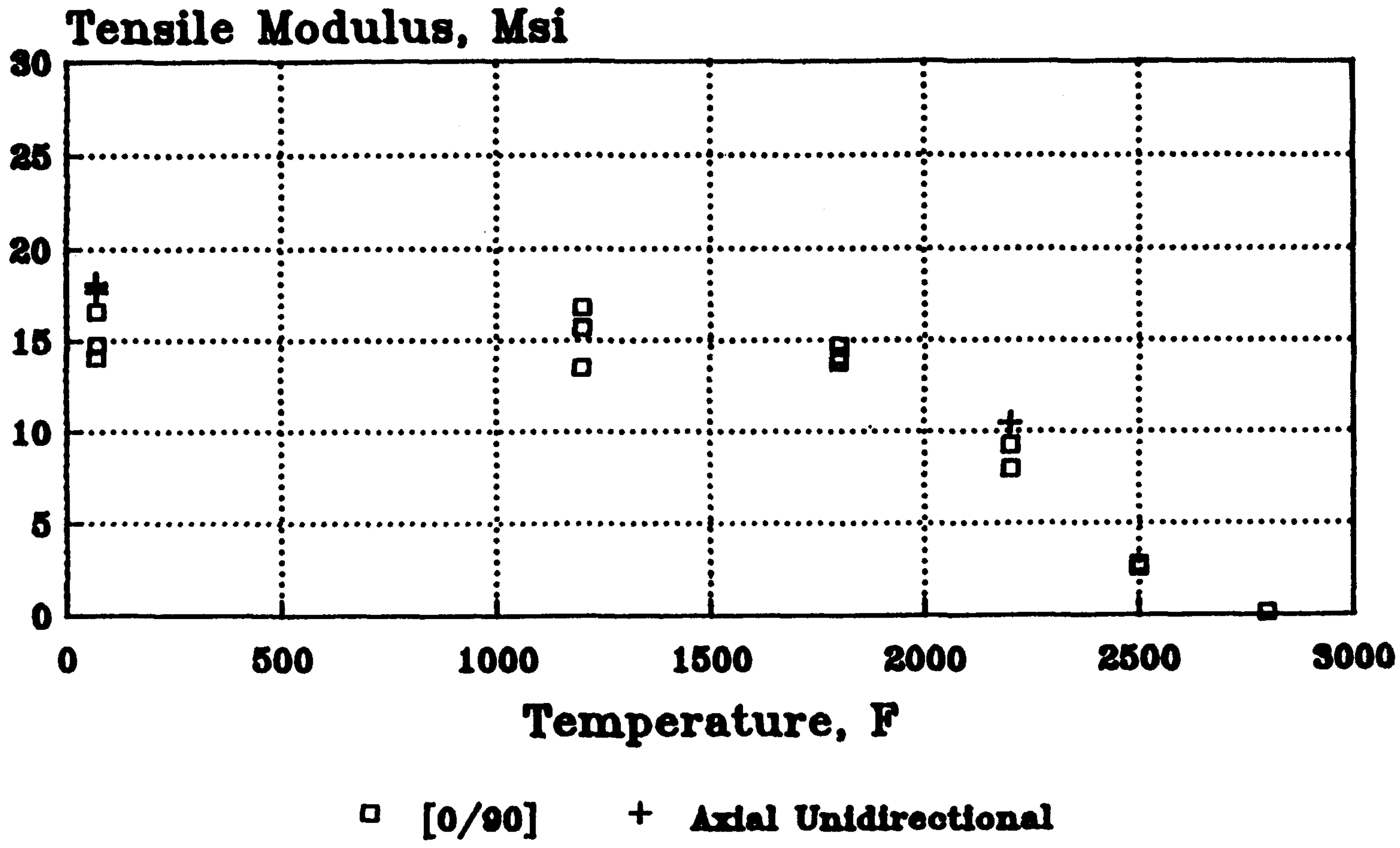
Tensile Stress–Strain Response of Unidirectional Nicalon/CAS



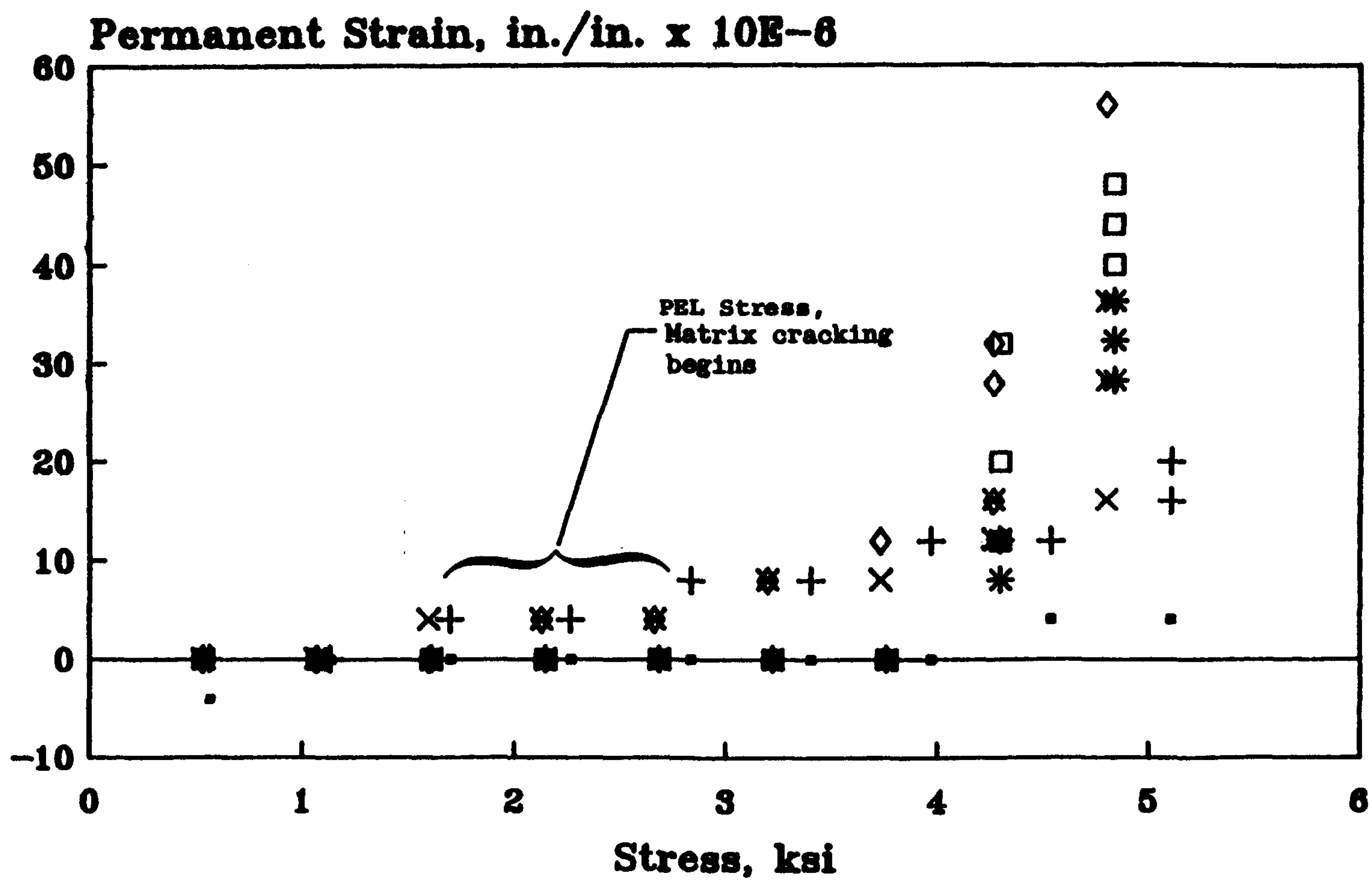
Tensile Strength vs. Temperature
Nicalon/CAS CMC



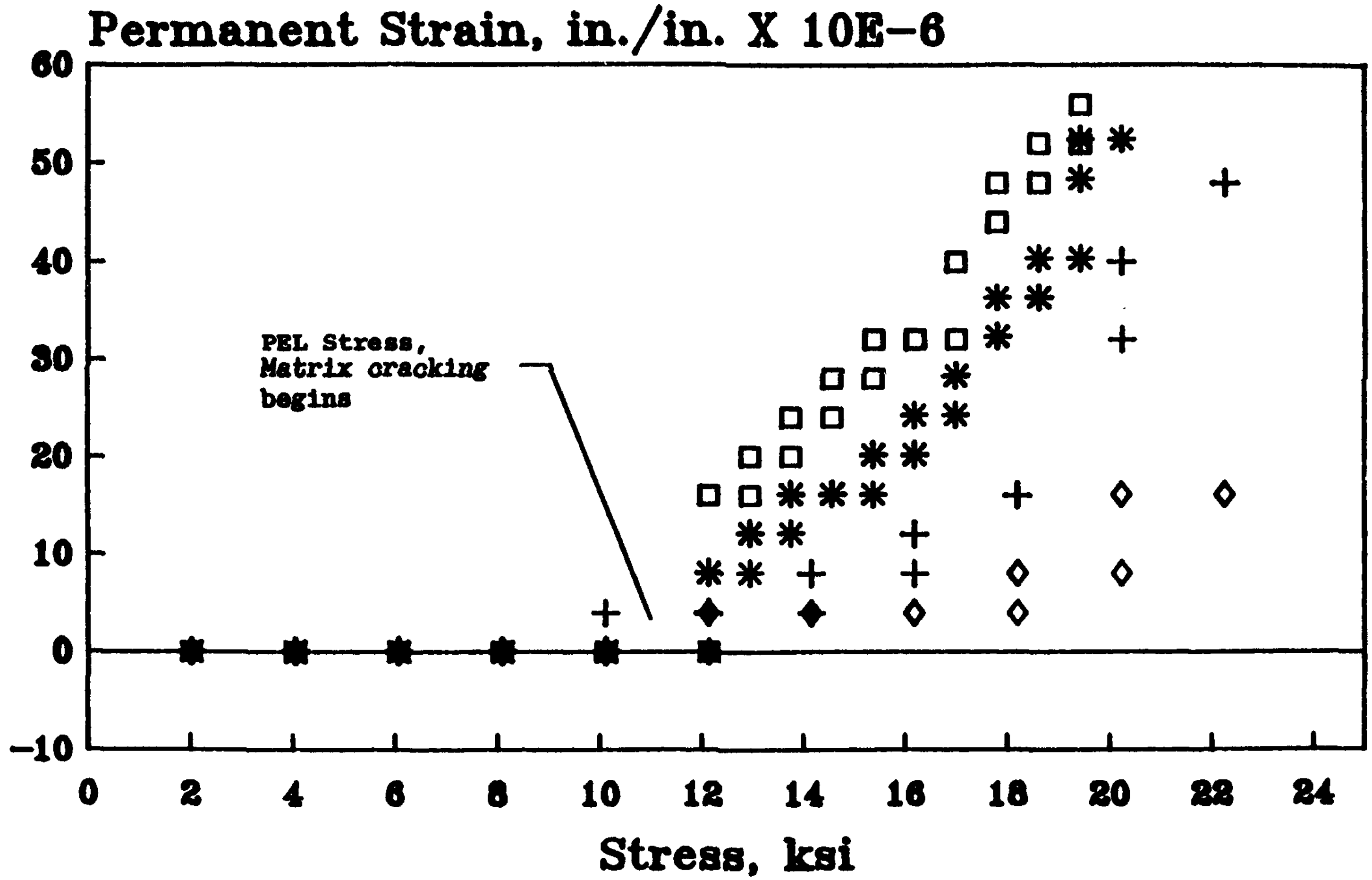
Tensile Modulus vs. Temperature
Nicalon/CAS CMC



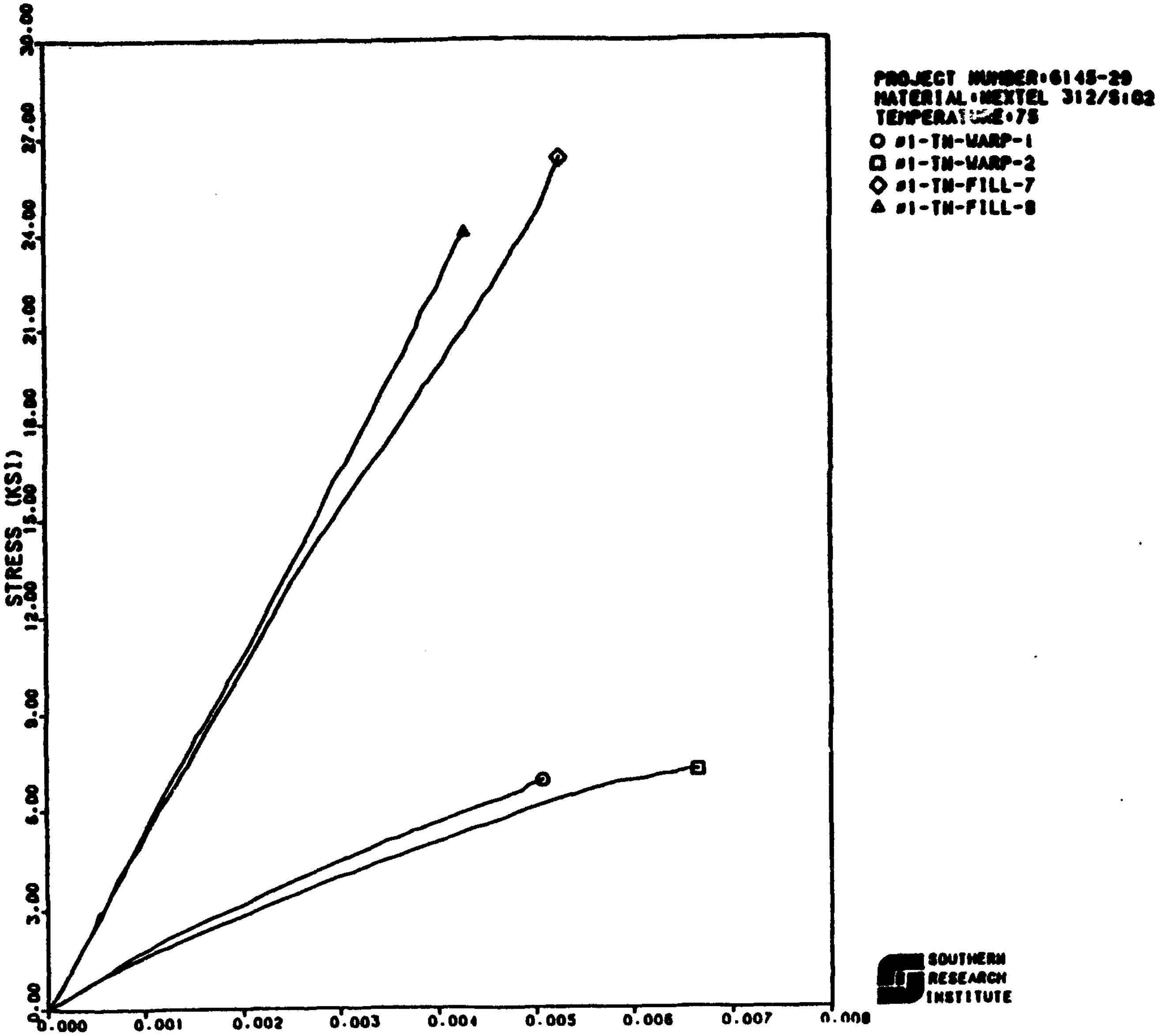
Permanent Strain vs. Stress
[0/90] Nicalon/CAS CMC



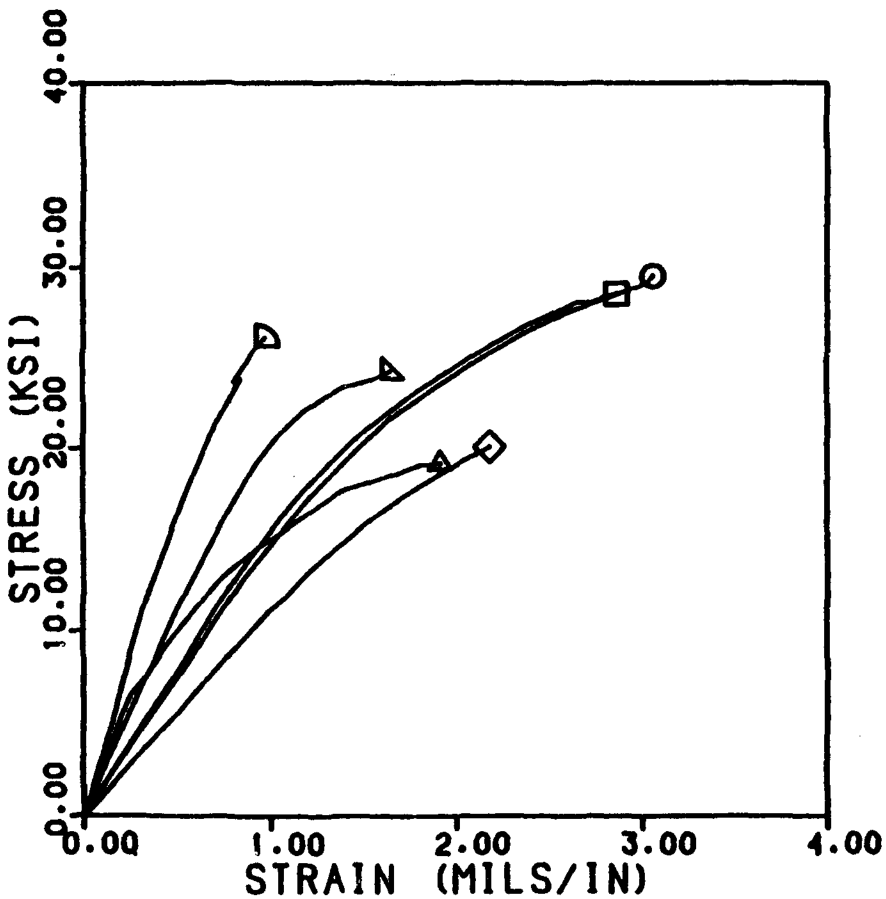
Permanent Strain vs. Stress
Unidirectional Nicalon/CAS CMC



Tensile Stress–Strain Response of PWA Nextel 312/SiO2 Angle–Interlock

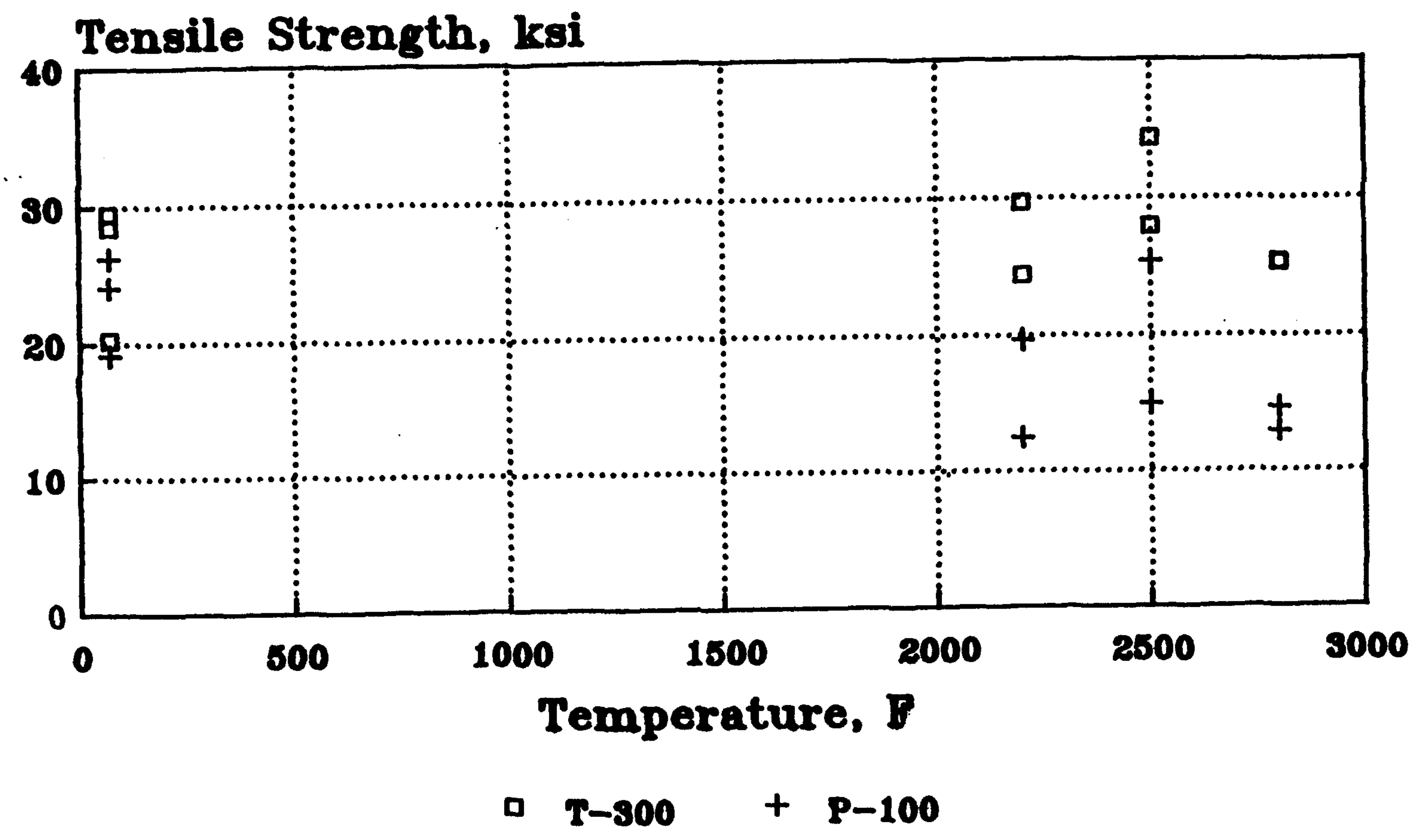


RCI P–100 and T–300 2DCC @ Room Temperature

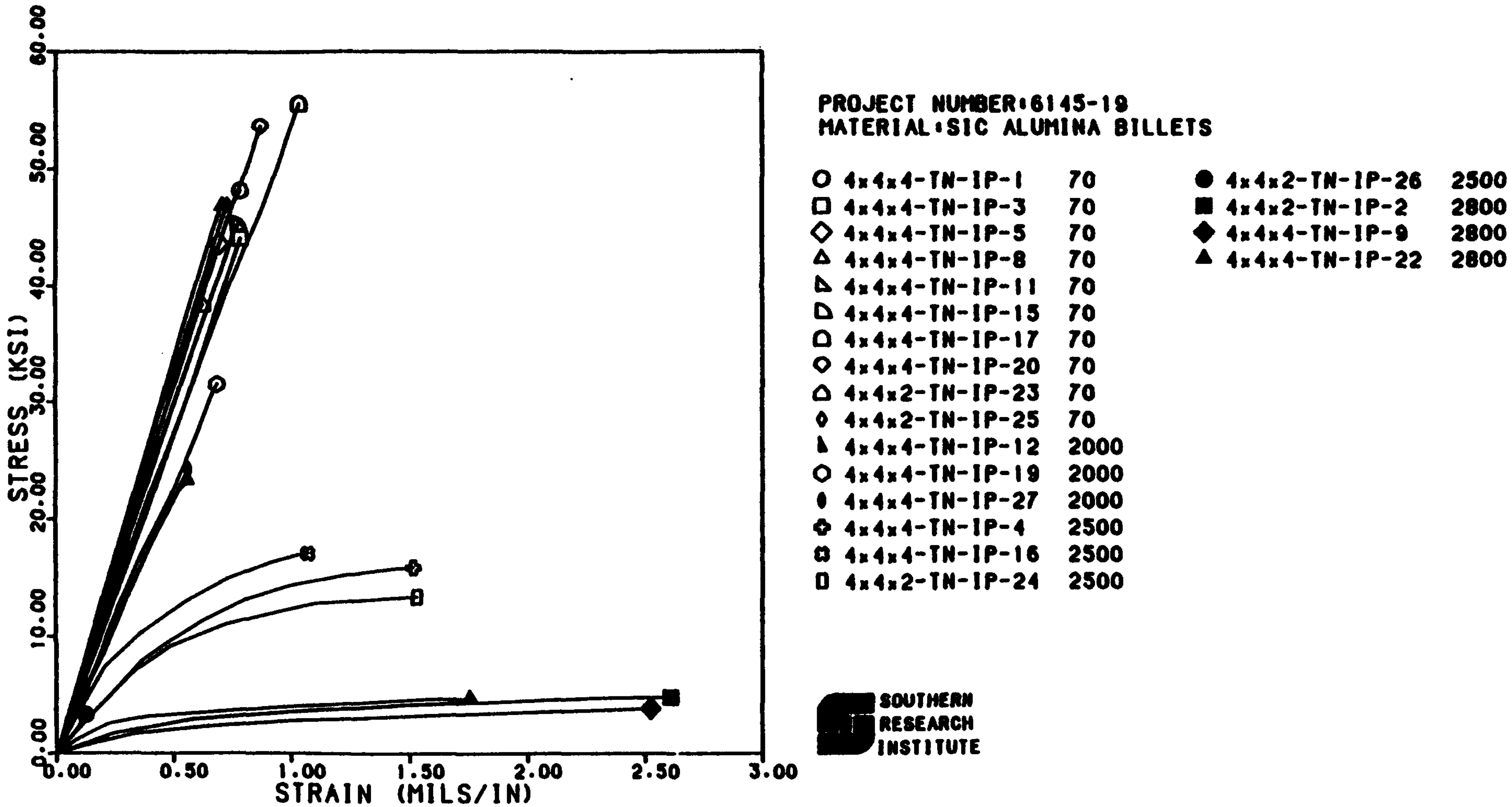


SOUTHERN RESEARCH INSTITUTE

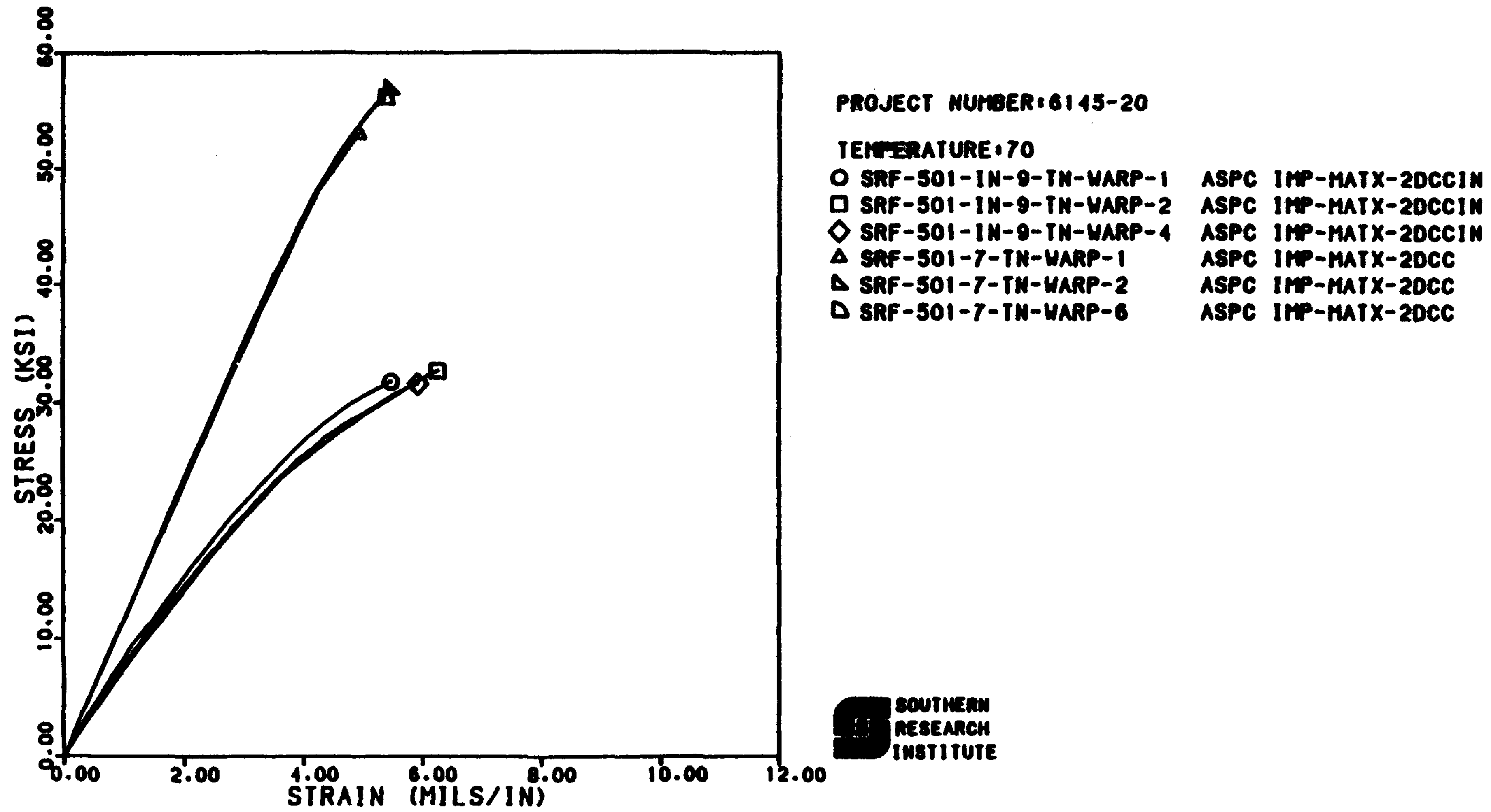
Tensile Strength vs. Temperature for RCI P-100 & T-300 2DCC



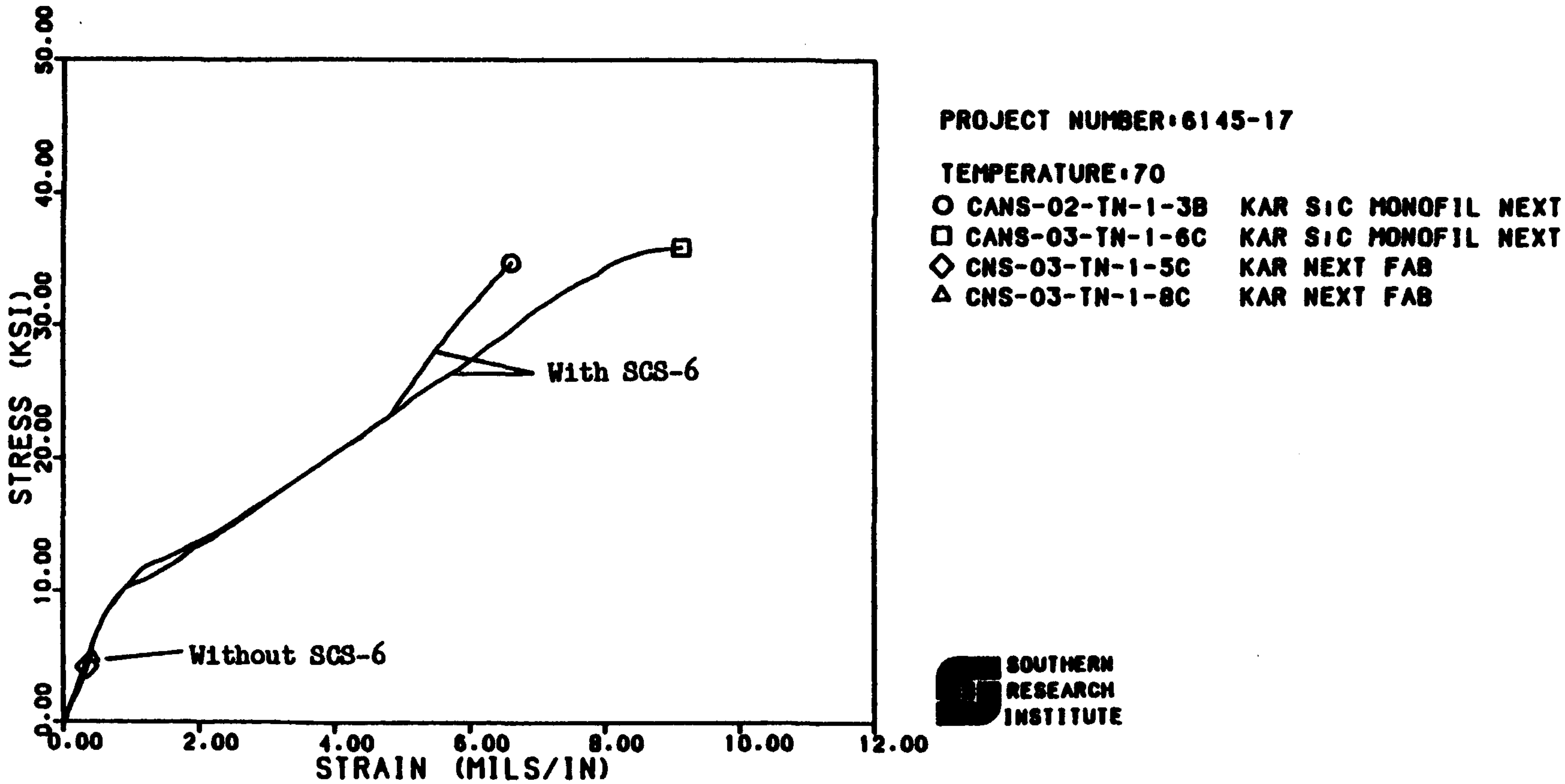
Arco SiC Whisker Reinforced Alumina
@ RT, 2000, 2500, 2800 F



ASPC Inhibited and Uninhibited 2DCC's @ Room Temperature



Kaiser Nextel/SiC with and w/o SCS–6 Monofilament
@ Room Temperature



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